A COMPLEX PERMITTIVITY AND PERMEABILITY MEASUREMENT SYSTEM FOR ELEVATED TEMPERATURES

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I. Introduction

This research is being supported by Grant No. NAG 3-972 from NASA Lewis Research Center (LeRC). The technical monitor is Mr. C. A. Stearns of the Environmental Durability Branch. The goals of this research program are threefold:

1) To fully develop a method to measure the permittivity and permeability of special materials as a function of frequency in the range of 2.6 to 18 GHz, and of temperature in the range of 25 to 1100° C; 2) To assist LeRC in setting up an in-house system for the measurement of high-temperature permittivity and permeability; 3) To measure the complex permittivity and permeability of special materials as a function of frequency and temperature to demonstrate the capability of the method.

II. Project Progress

A. Background

The method chosen for characterizing the sponsorfurnished materials is based on standards issued by the American Society for Testing and Materials (ASTM), standards D 2520-86 [1] (Complex Permittivity of Solid Electrical and Frequencies Materials Microwave at Insulating Temperatures to 1650°C) and F 131-70 [2] (Complex Dielectric Constant of Nonmetallic Magnetic Materials at Microwave This method relies on perturbation of a Frequencies). resonant cavity with a small volume of sample material. Different field configurations in the cavity can be used to separate electric and magnetic effects. Moore, et al. [3] presented a detailed explanation of this technique at the December, 1987 International Symposium on Infrared and MMW Technology, with particular emphasis on applications with anisotropic ferrites. An updated version of that paper has been accepted for publication in the American Institute of Physics (AIP) Review of Scientific Instruments.

Figure 1 illustrates the physical configuration of the waveguide cavity and sample. The cavity consists of a section of rectangular waveguide terminated at each end with In the center of one wall is a small a vertical slot iris. hole through which the sample is introduced. permittivity measurements the hole is in the center of the broad wall, and an odd resonance mode (i.e., TE10n, with n The sample is thus located at a point of odd) is used. maximum electric field, and for small sample volumes the field is nearly constant over the sample region. Similarly, for permeability measurements the sample hole is located in the middle of the narrow wall and an even resonance mode is Thus, the sample is at a point of maximum magnetic used. field.

Typically, the sample is contained in a small bore quartz tube. Such tubes have been used with powdered samples, fiber samples, and thin ceramic rods. A calibration measurement for such a sample would include measurement of the cavity with an empty quartz tube in place, so that perturbation effects could be solely attributed to the sample.

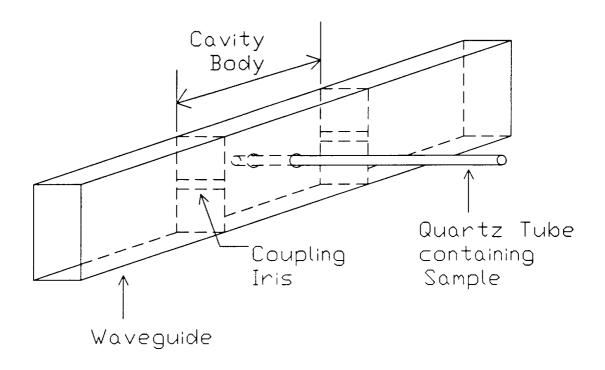


Figure 1. Diagram showing configuration of rectangular waveguide cavity used for measurements. The illustrated orientation of the sample would be used with odd modes for permittivity measurements. For permeability measurements, the sample would be inserted parallel to the broad wall.

B. Cavity Design

Drawings of a waveguide cavity for use at X band have been furnished separately to LeRC. Cavities for other bands are similar except for size. Key features of the cavity design include the location of sample holes as explained above; the material from which the assembly is fabricated; the length of the cavity; and the location of the joint between pieces. The assembly is fabricated from Hastelloy, an alloy of nickel developed to withstand temperatures in excess of 1200° C. Cavity lengths are designed to support three modes of the form TE_{10n}, so that each cavity will have either two odd modes and one even, or two even modes and one It is expected that the complete system will include two cavities, one of each type, in each band. cavities with two odd modes can be joined at a seam through the narrow walls, while those cavities with two even modes can be joined at a seam through the broad walls. of the seam in the narrow wall will minimize its effect on permittivity measurements, while a seam in the broad wall is best for permeability measurements. Table I shows possible cavity lengths for each waveguide band, along with the inband resonant modes which would be expected for each length. The width and height of each cavity are assumed to be the dimensions of the standard rectangular waveguide for each band, i.e., WR-187 for C band, WR-137 for Xn band, WR-90 for X band, and WR-62 for Ku band.

TABLE I
RESONANT MODE VS FREQUENCY (GHZ) FOR VARIOUS LENGTHS

Mode:	TE ₁₀₂	TE ₁₀₃	TE ₁₀₄	^{TE} 105	TE ₁₀₆		
<u>C band</u>							
6.6"		4.1393	4.7676	5.47046			
4.8"	3.9980	4.8520	5.8415				
Xn band							
4.3"		5.9543	6.9741	8.0988			
5.5"			6.0764	6.8763	7.7426		
X band							
3.3"		8.4722	9.7039	11.0882			
3.8"			9.0325	10.1633	11.3939		
<u>Ku band</u>							
2.1"		12.692	14.710	16.954			
2.6"			13.132	14.792	16.598		

C. Room Temperature Measurements

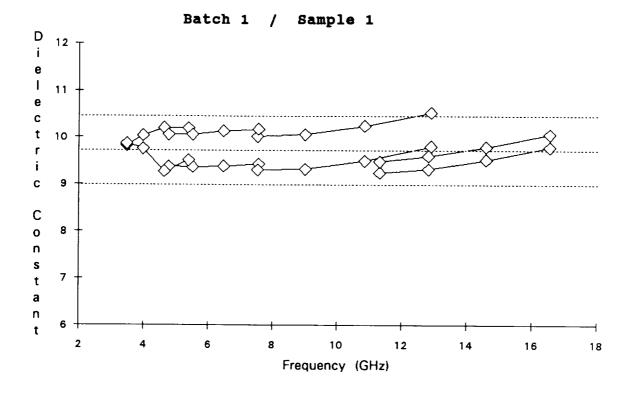
The third goal of this program is to demonstrate the capabilities of this method by applying it to special samples provided by NASA Lewis. Eight samples were sent in the initial batch, five of which arrived intact. The other three were broken and mixed together. We were unable to distinguish the pieces by composition and reunite them into measurable samples, so they will be put aside and returned to NASA after the other samples are measured. These samples have been labelled Batch 1. Two other batches have also been received, under the designations 60-1015 and 60-1520. They will be referred to as batches 2 and 3, respectively. All unbroken samples in the three batches have been measured at room temperature.

Room temperature measurements were performed in waveguide cavities which were either made of copper (C and Xn bands) or gold-plated (X and Ku bands). These materials provide a higher conductivity and, consequently, better quality factor in the cavities than the nickel which is required for higher temperatures. A higher quality factor makes smaller changes distinguishable, and thus makes the measurements more sensitive.

Results from the room temperature measurements are presented two different ways. One set of plots presents the with average and standard deviation superimposed; the other set of plots contains error bars about each measured point. Each sample was measured twice in each of the four frequency bands: C (4-6 GHz), Xn (6-8 GHz), X (8.4-12.4 GHz), and Ku (12.4-18.0 GHz). contains data from one sample, and each line segment on the plot represents one set of measurements in one cavity. of the plots thus contain eight line segments (four bands times two measurements). Within the cavity for each frequency band, typically eight different resonant modes were obtainable, four odd modes and four even modes. complex permittivity measurements, each sample was inserted parallel to the E-field in the cavity and the four odd modes used; for complex permeability measurements each sample was inserted perpendicular to the E-field in the (parallel to the broad wall of the waveguide) and the four even modes used. Thus each line segment on a plot will contain four data points corresponding to four different resonant frequencies. (Permittivity results permeability results are presented in separate sets of The resonant frequencies at the edges of the bands plots.) between cavities sometimes overlapped. This is because some measured resonances were outside the customary limits of the waveguide band. All measured resonances were well above cutoff and below the cutoff frequency for the next higher mode, however.

Each sample was measured two times in each band. When the sample was long enough, the two measurements were performed at different locations along the length of the sample. In several cases the sample was not long enough to extend through the entire cavity. Because the volume of sample inside the cavity was indeterminate, it was not possible to perform meaningful dielectric calculations in those cases. Thus, measurements of samples 1-2 in the first batch; samples 1-5 of the second batch; and samples 1-3 of the third batch, at even modes (parallel to the broad wall) in the C-band cavity are not included. Also, samples 1-2 of the third batch and sample 4 of the second batch are not characterized at even modes in the Xn band cavity.

Figures 2-6 are plots of the calculated dielectric constant and loss tangent of the five surviving samples in batch 1; Figures 7-11 show calculated dielectric constants and loss tangents for the five samples in batch 2; and 12-19 show the dielectric constants tangents for the eight samples of batch 3. All plots are different line with the frequency, representing individual measurements in a single cavity, as The three dotted lines superimposed on explained above. each plot represent an average value with one standard The average was taken over all deviation on either side. frequency values and all samples in a batch. (Thus the average and standard deviation lines are the same on all plots of a given batch.) The average was taken across all frequencies because normal dielectric behavior of ceramic materials is not frequency dependent in the microwave regions. These averages are compiled in Table 2.



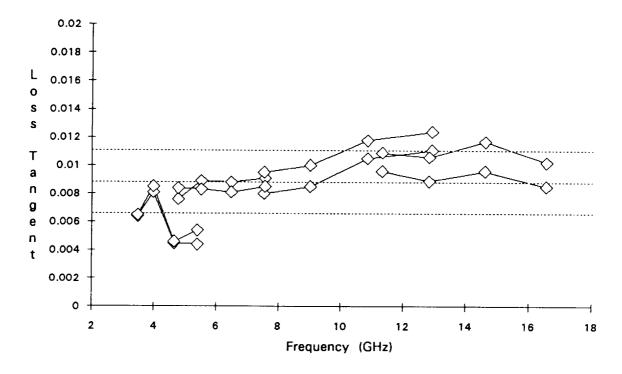
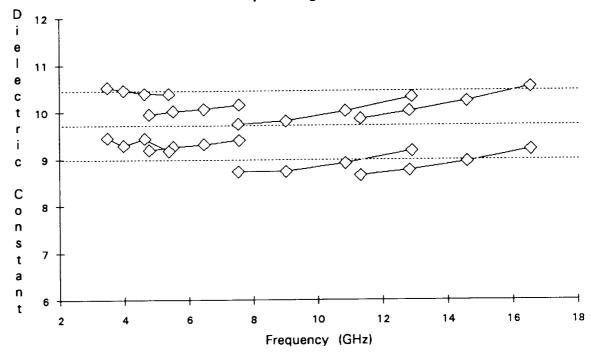


Figure 2. Data from two measurements at each of four frequency bands is plotted vs frequency (solid lines). The average value over all five samples, sixteen frequency points, and two measurements is superimposed along with one standard deviation above and below the average (dotted lines).

Batch 1 / Sample 2



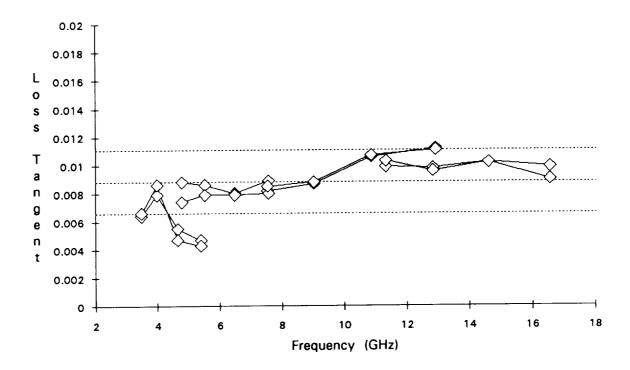
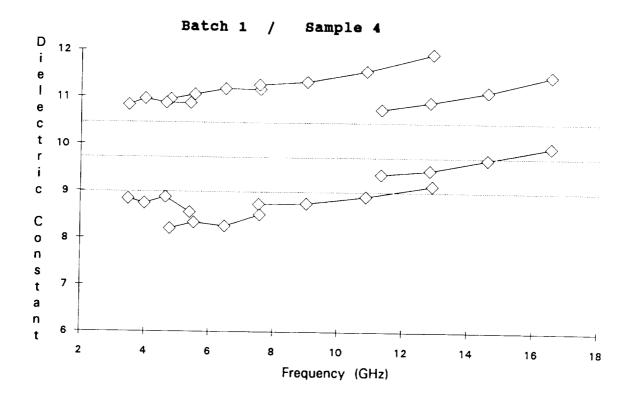


Figure 3. Data from two measurements at each of four frequency bands is plotted vs frequency. (solid lines) The average value over all five samples, sixteen frequency points, and two measurements is superimposed along with one standard deviation above and below the average (dotted lines).



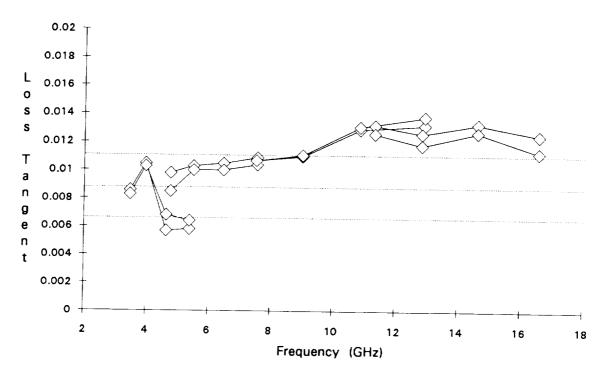
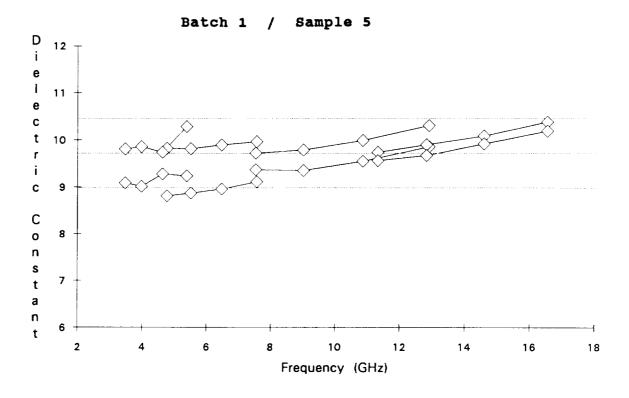


Figure 4. Data from two measurements at each of four frequency bands is plotted vs frequency (solid lines). The average value over all five samples, sixteen frequency points, and two measurements is superimposed along with one standard deviation above and below the average (dotted lines).



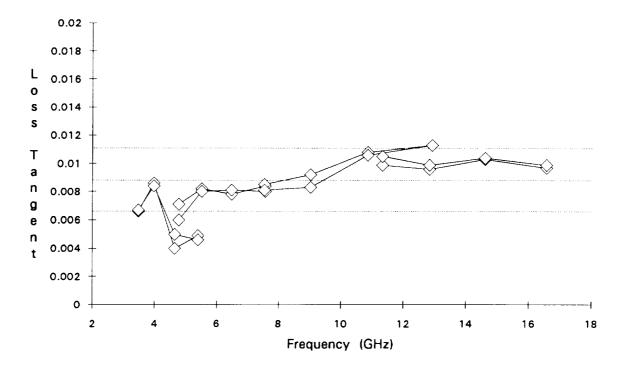
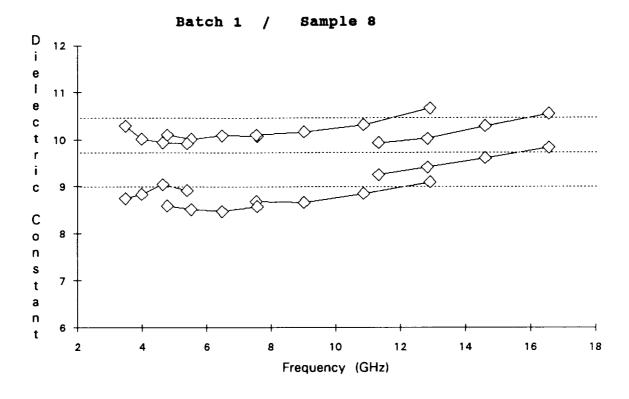


Figure 5. Data from two measurements at each of four frequency bands is plotted vs frequency (solid lines. The average value over all five samples, sixteen frequency points, and two measurements is superimposed along with one standard deviation above and below the average (dotted lines).



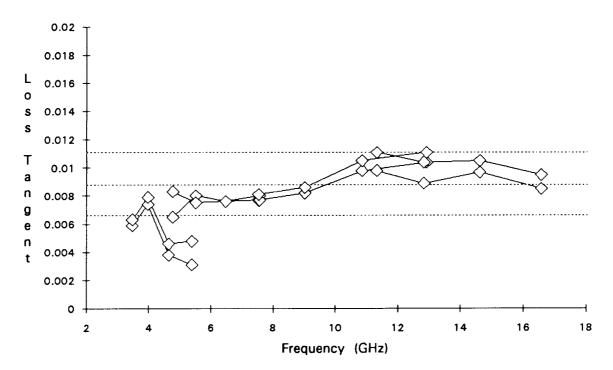


Figure 6. Data from two measurements at each of four frequency bands is plotted vs frequency (solid lines). The average value over all five samples, sixteen frequency points, and two measurements is superimposed along with one standard deviation above and below the average (dotted lines).

Batch 2 / Sample 1 12 е 11 е С t 10 i С 9 C 8 0 n s 7 t а n t 16 18 12 2 Frequency (GHz)

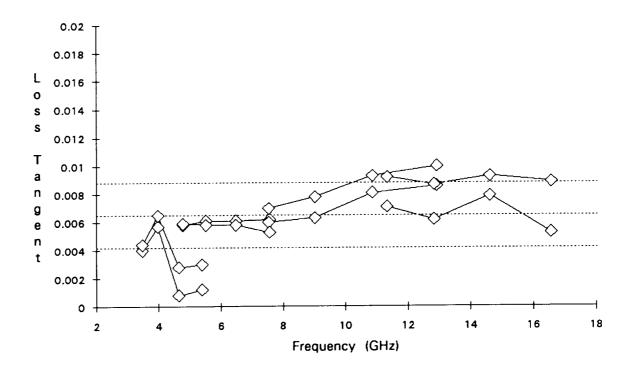
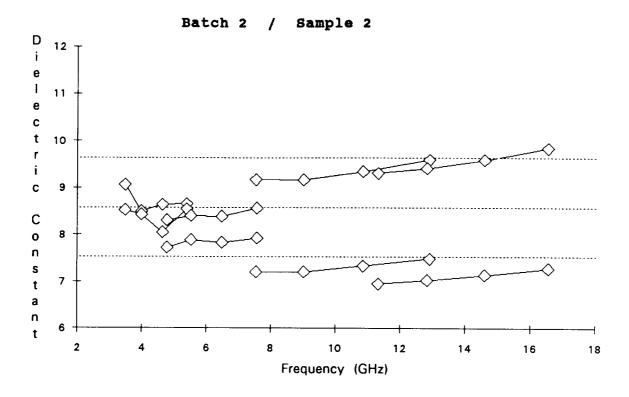


Figure 7. Data from two measurements at each of four frequency bands is plotted vs frequency (solid lines). The average value over all five samples, sixteen frequency points, and two measurements is superimposed along with one standard deviation above and below the average (dotted lines).



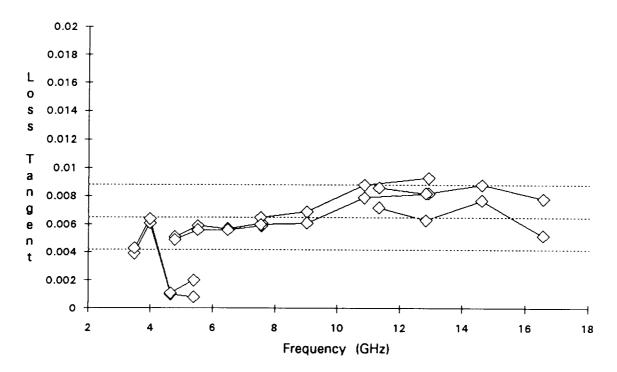
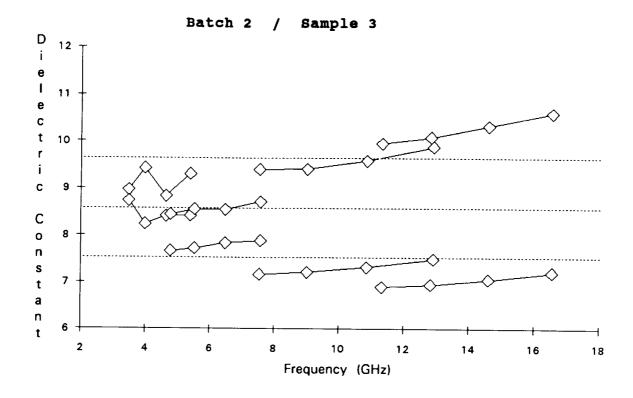


Figure 8. Data from two measurements at each of four frequency bands is plotted vs frequency (solid lines). The average value over all five samples, sixteen frequency points, and two measurements is superimposed along with one standard deviation above and below the average (dotted lines).



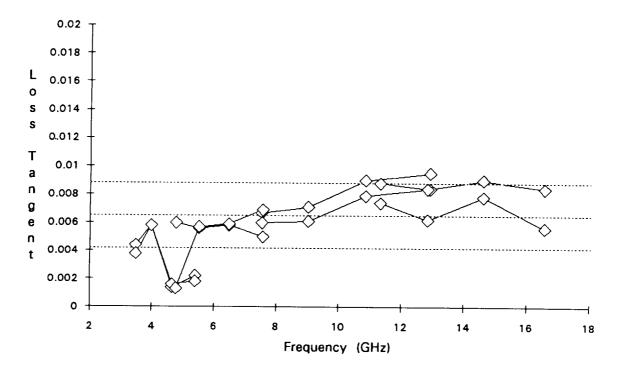
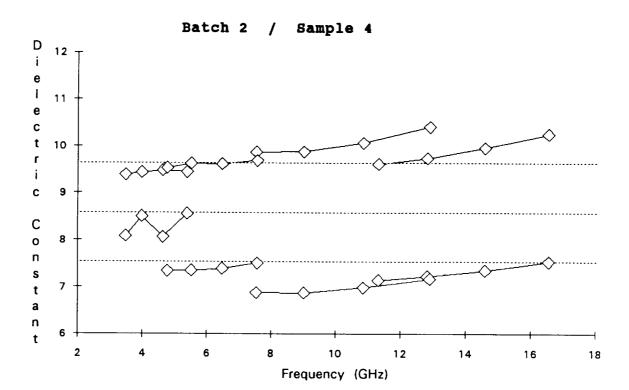


Figure 9. Data from two measurements at each of four frequency bands is plotted vs frequency (solid lines). The average value over all five samples, sixteen frequency points, and two measurements is superimposed along with one standard deviation above and below the average (dotted lines).



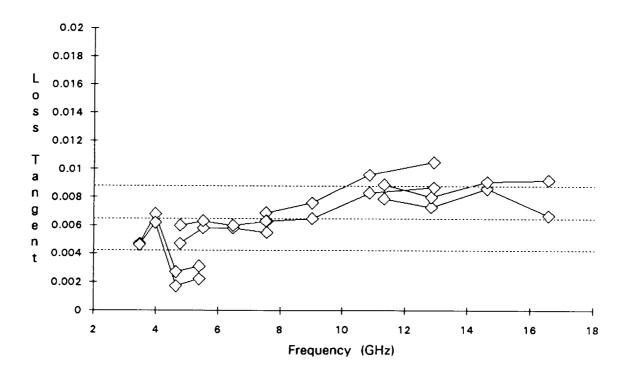


Figure 10. Data from two measurements at each of four frequency bands is plotted vs frequency (solid lines). The average value over all five samples, sixteen frequency points, and two measurements is superimposed along with one standard deviation above and below the average (dotted lines).

Batch 2 Sample 5 12 е 1 11 е С t 10 r i С 9 C 8 0 n S 7 t а n 6 t 2 10 12 14 16 18

Frequency (GHz)

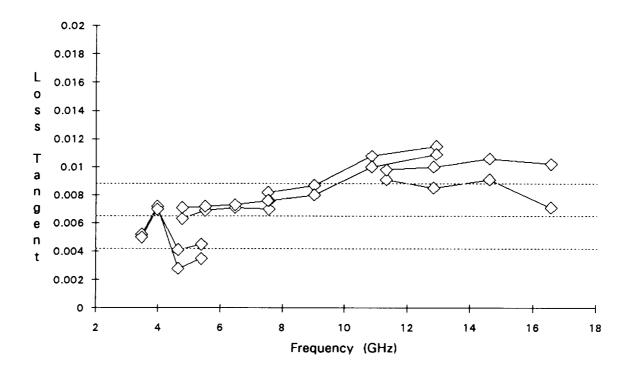
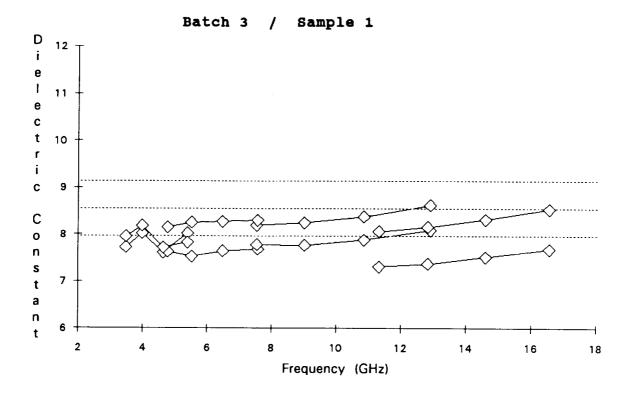


Figure 11. Data from two measurements at each of four frequency bands is plotted vs frequency (solid lines). The average value over all five samples, sixteen frequency points, and two measurements is superimposed along with one standard deviation above and below the average (dotted lines).



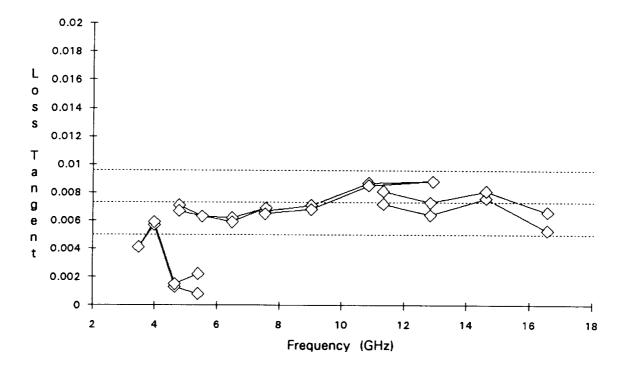
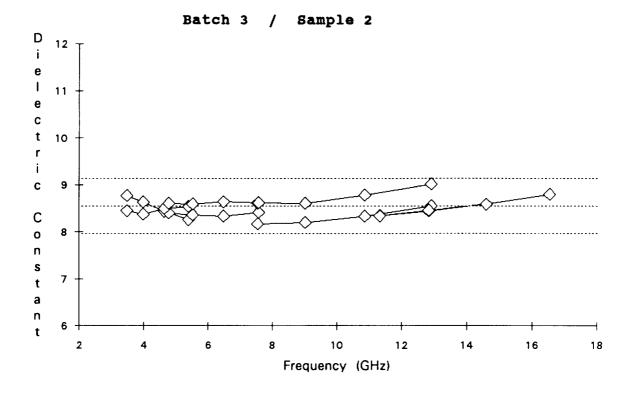


Figure 12. Data from two measurements at each of four frequency bands is plotted vs frequency (solid lines). The average value over all eight samples, sixteen frequency points, and two measurements is superimposed along with one standard deviation above and below the average (dotted lines).



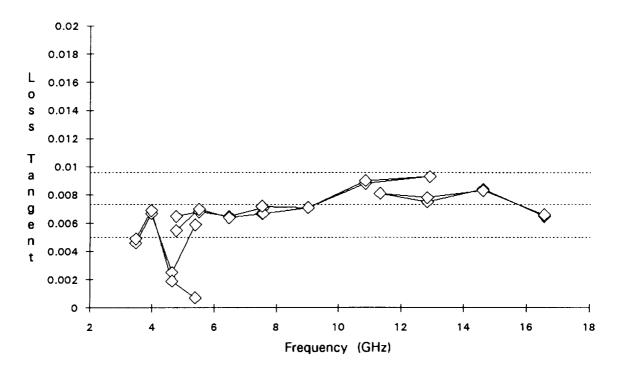
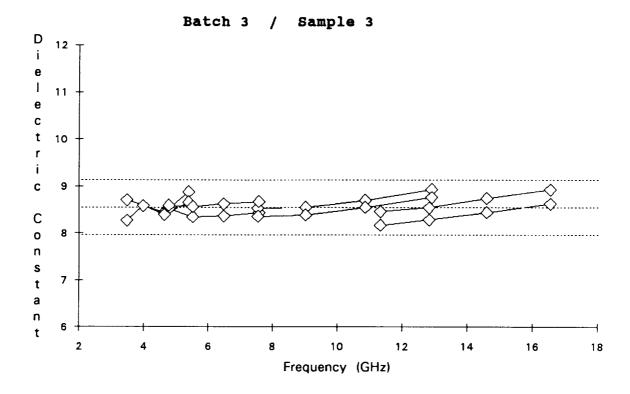


Figure 13. Data from two measurements at each of four frequency bands is plotted vs frequency (solid lines). The average value over all eight samples, sixteen frequency points, and two measurements is superimposed along with one standard deviation above and below the average (dotted lines).



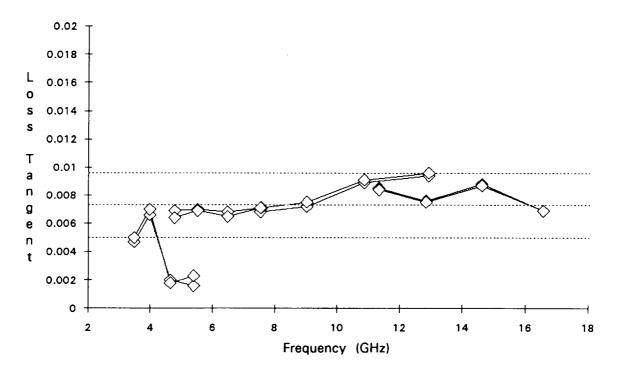
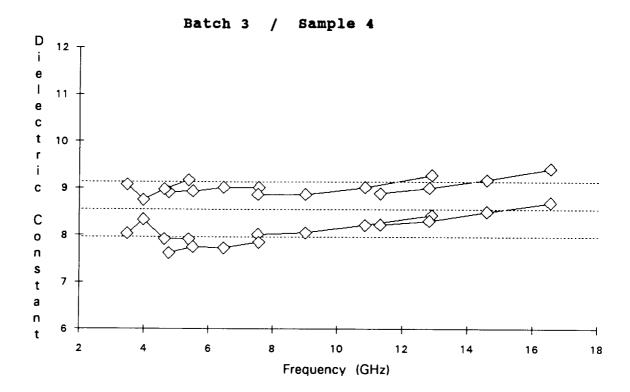


Figure 14. Data from two measurements at each of four frequency bands is plotted vs frequency (solid lines). The average value over all eight samples, sixteen frequency points, and two measurements is superimposed along with one standard deviation above and below the average (dotted lines).



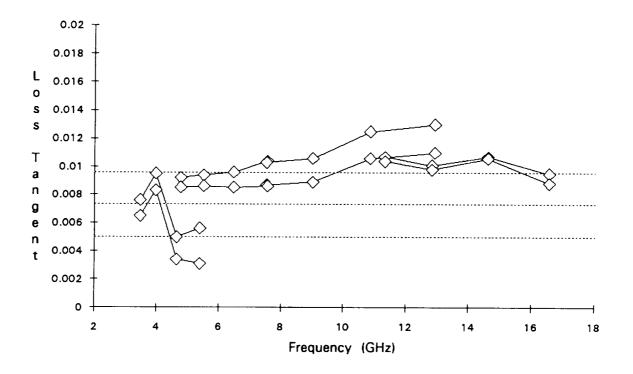
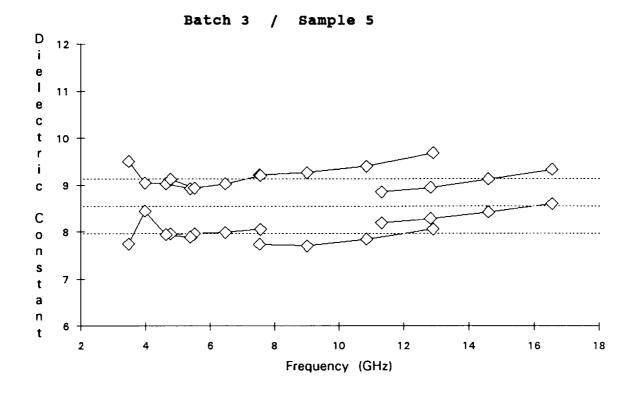


Figure 15. Data from two measurements at each of four frequency bands is plotted vs frequency (solid lines). The average value over all eight samples, sixteen frequency points, and two measurements is superimposed along with one standard deviation above and below the average (dotted lines).



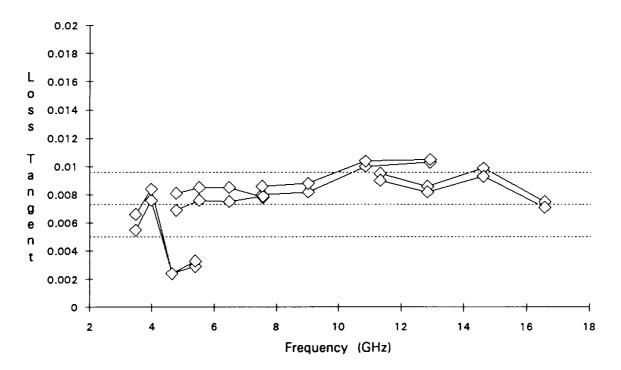
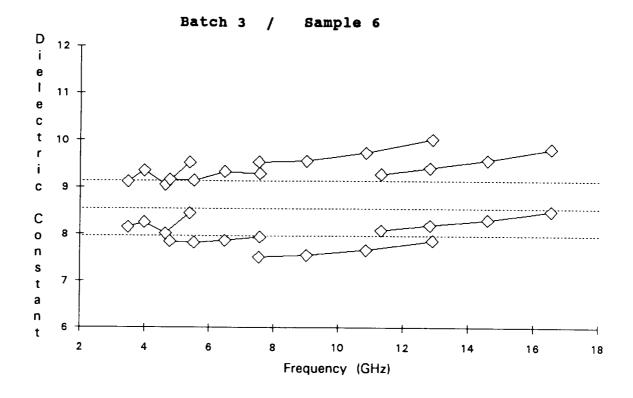


Figure 16. Data from two measurements at each of four frequency bands is plotted vs frequency (solid lines). The average value over all eight samples, sixteen frequency points, and two measurements is superimposed along with one standard deviation above and below the average (dotted lines).



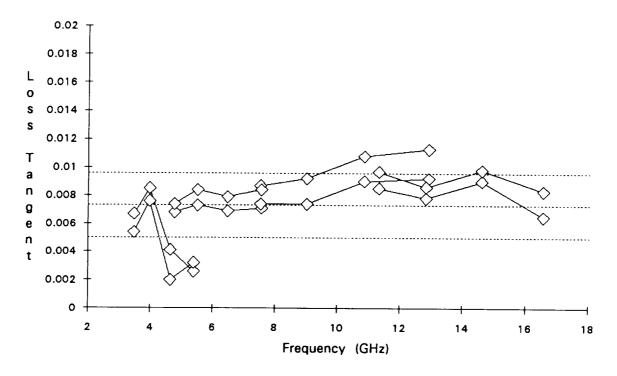
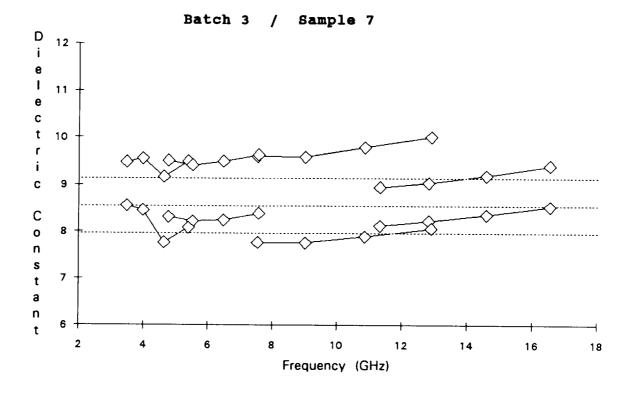


Figure 17. Data from two measurements at each of four frequency bands is plotted vs frequency (solid lines). The average value over all eight samples, sixteen frequency points, and two measurements is superimposed along with one standard deviation above and below the average (dotted lines).



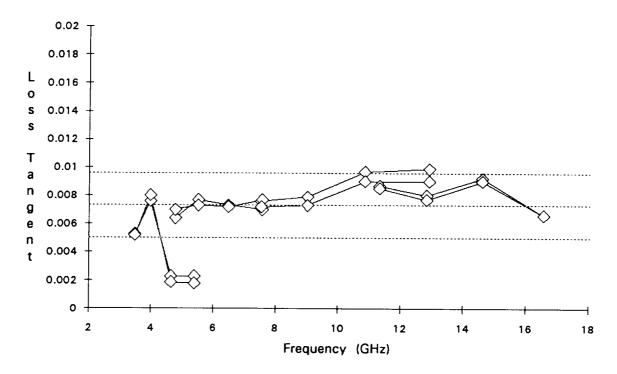
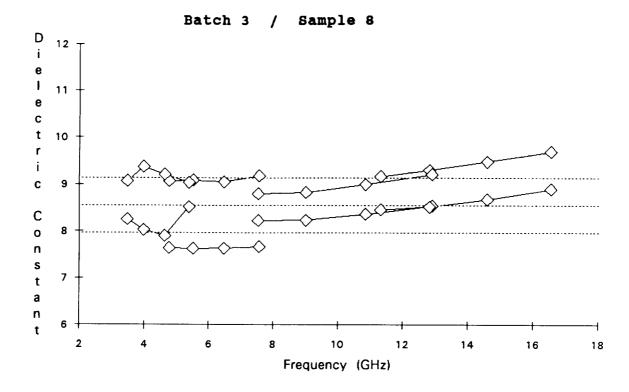


Figure 18. Data from two measurements at each of four frequency bands is plotted vs frequency (solid lines). The average value over all eight samples, sixteen frequency points, and two measurements is superimposed along with one standard deviation above and below the average (dotted lines).



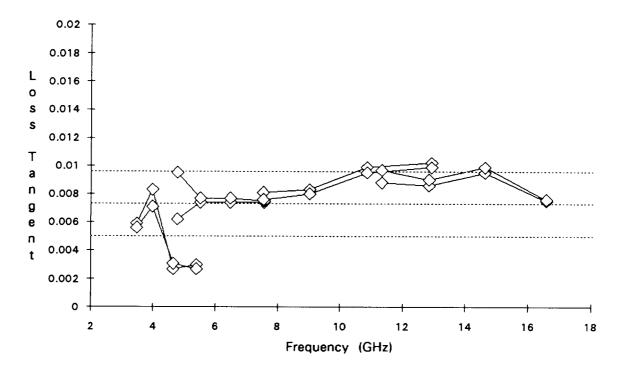


Figure 19. Data from two measurements at each of four frequency bands is plotted vs frequency (solid lines). The average value over all eight samples, sixteen frequency points, and two measurements is superimposed along with one standard deviation above and below the average (dotted lines).

TABLE 2
AVERAGE DIELECTRIC VALUES FOR EACH SAMPLE BATCH

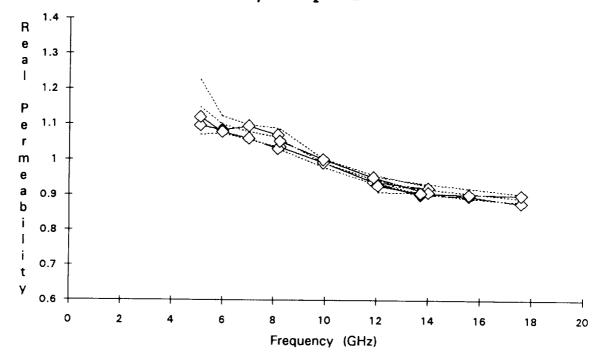
Batch	Average Dielectric Constant	Standard Deviation	Average Loss Tangent	Standard Deviation
1	9.72	.73	.0088	.0022
2	8.58	1.05	.0065	.0023
3	8.55	. 58	.0073	.0023

In Figures 20-24, the real and imaginary parts of the complex magnetic permeability of the samples of batch 1 are plotted versus frequency. Figures 25-29 represent the results of measurements of batch 2 samples, and Figures 30-37 are from the samples of batch 3. For the real and imaginary permeability plots, average and standard deviation values are again represented with dotted lines. This time, however, the average is taken only over the samples of a given batch at a particular frequency. Since typical magnetic properties often show frequency dependence in the microwave regions, each mode was averaged separately. of these plots do not contain line segments from the C band cavity, or in a few instances from the Xn band cavity. explained above, this is because the samples in these cases were shorter than the broad-wall dimension of the waveguide.

One characteristic worth noting in the plots is a sometimes wide disparity between multiple measurements of the same sample. This is most likely due to inhomogeneity in the samples. As noted above, these measurements were performed on different sections of each sample rod whenever possible. In addition, since more of each sample was present in the larger cavity volumes of the lower frequency bands, measurements at the lower bands have the effect of averaging over more of the sample material. For this

reason, higher frequency cavities will be more sensitive to inhomogeneities in the samples when different regions of the sample are measured. Unfortunately, the same section was not always measured in all four bands. Thus, traces from band to band do not always represent the same section of material; hence they would not necessarily "line up". Finally, for most of the permittivity measurements, especially in the cavities at Xn, X, and Ku bands, a slight upward slope with increasing frequency is exhibited by the data curves. This is due to the finite volume of the samples. A first order correction for sample volumes which do not vanish has already been applied in the analysis software, but residual effects manifest themselves as the slope present in most of these plots. The "true" value would be approximately in the middle of the segment from each band.

Batch 1 / Sample 1



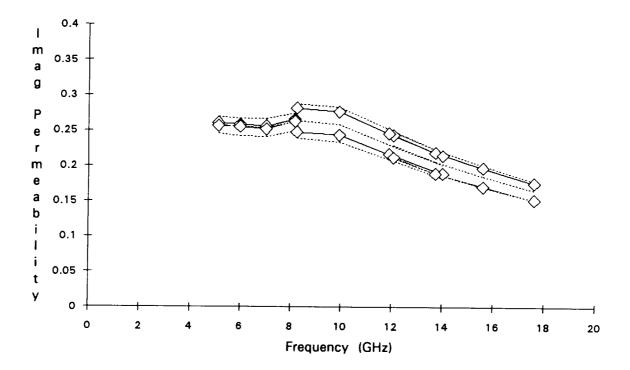
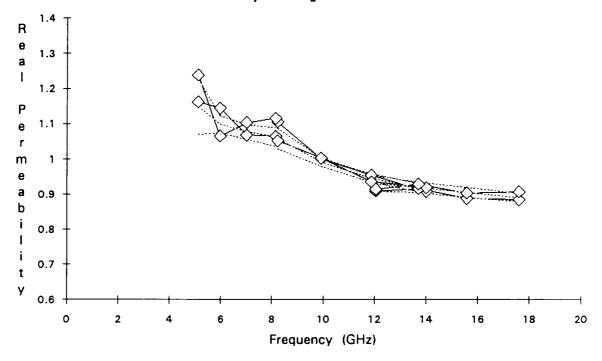


Figure 20. Data from two measurements at each of three frequency bands is plotted vs frequency (solid lines). The average value over all five samples and two measurements at each frequency point is superimposed along with one standard deviation above and below the average (dotted lines).

Batch 1 / Sample 2



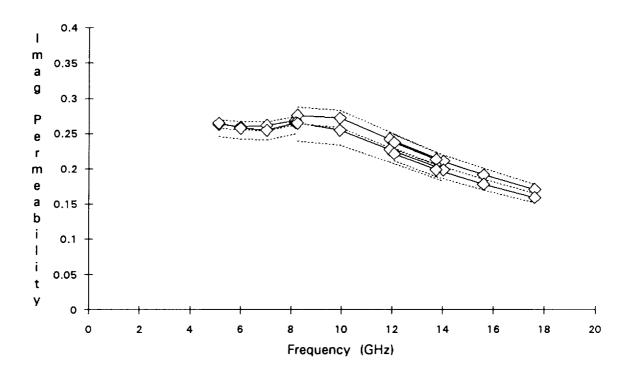
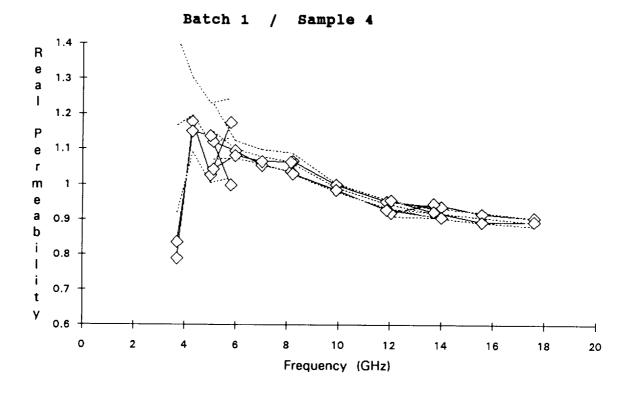


Figure 21. Data from two measurements at each of three frequency bands is plotted vs frequency (solid lines). The average value over all five samples and two measurements at each frequency point is superimposed along with one standard deviation above and below the average (dotted lines).



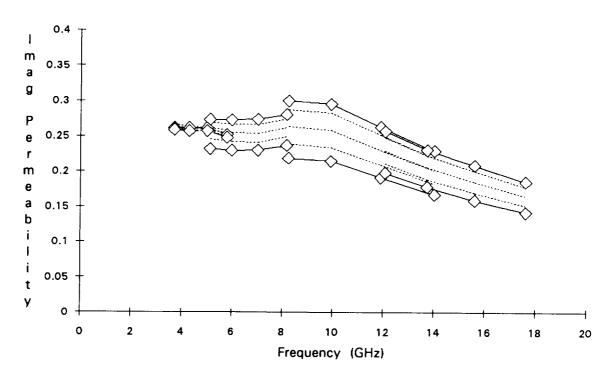
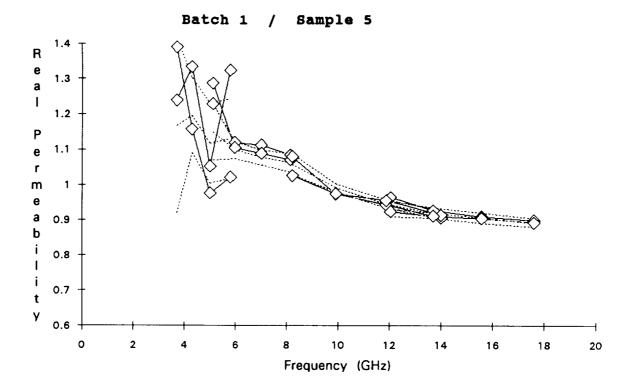


Figure 22. Data from two measurements at each of four frequency bands is plotted vs frequency (solid lines). The average value over all five samples and two measurements at each frequency point is superimposed along with one standard deviation above and below the average (dotted lines).



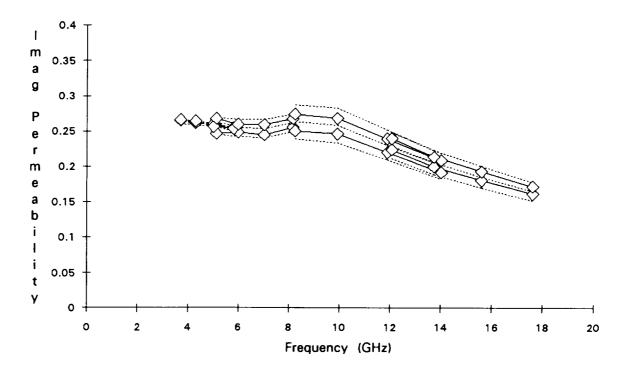
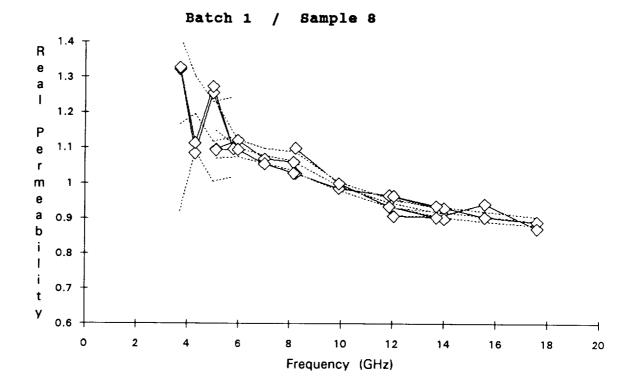


Figure 23. Data from two measurements at each of four frequency bands is plotted vs frequency (solid lines). The average value over all five samples and two measurements at each frequency point is superimposed along with one standard deviation above and below the average (dotted lines).



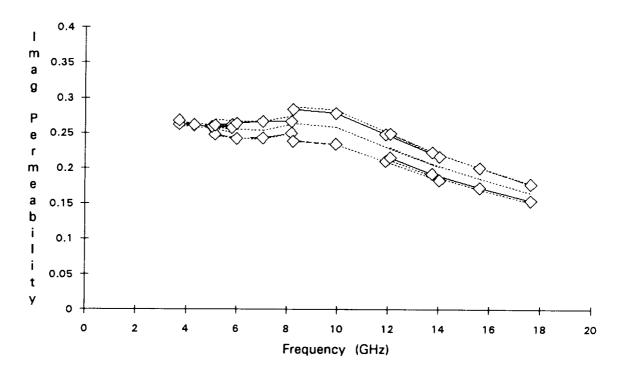
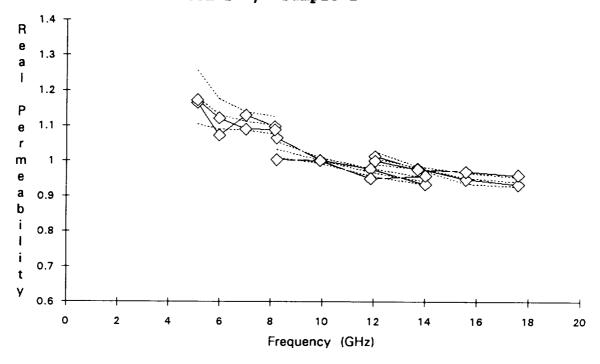


Figure 24. Data from two measurements at each of four frequency bands is plotted vs frequency (solid lines). The average value over all five samples and two measurements at each frequency point is superimposed along with one standard deviation above and below the average (dotted lines).

Batch 2 / Sample 1



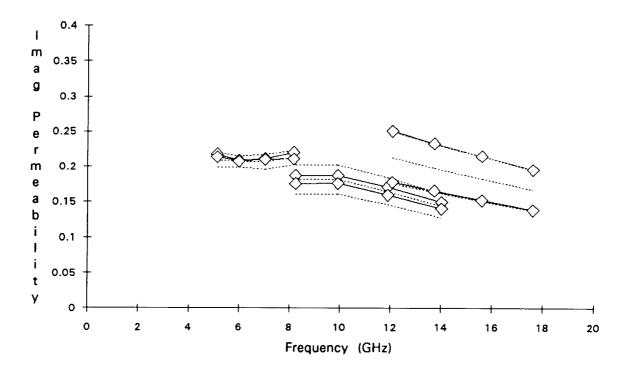
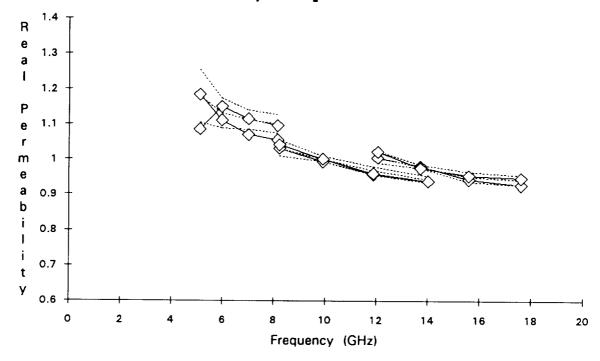


Figure 25. Data from two measurements at each of three frequency bands is plotted vs frequency (solid lines). The average value over all five samples and two measurements at each frequency point is superimposed along with one standard deviation above and below the average (dotted lines).



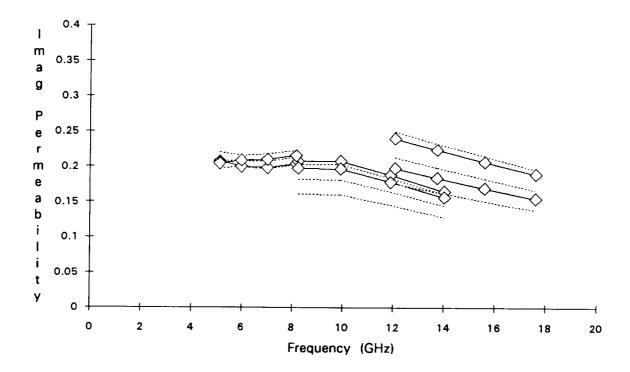
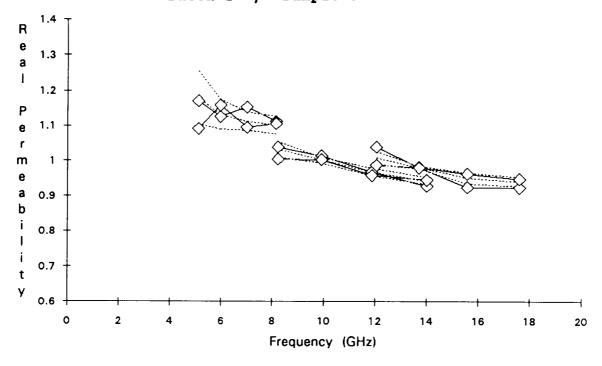


Figure 26. Data from two measurements at each of three frequency bands is plotted vs frequency (solid lines). The average value over all five samples and two measurements at each frequency point is superimposed along with one standard deviation above and below the average (dotted lines).



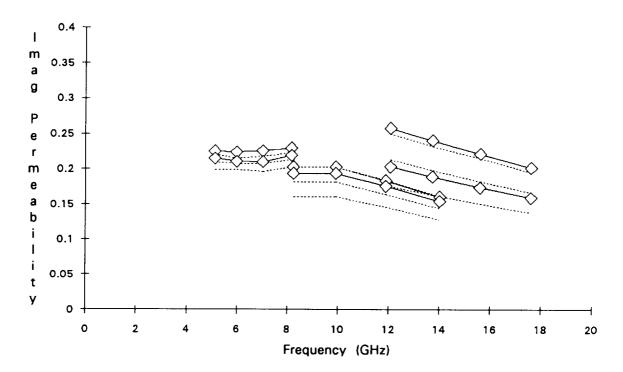
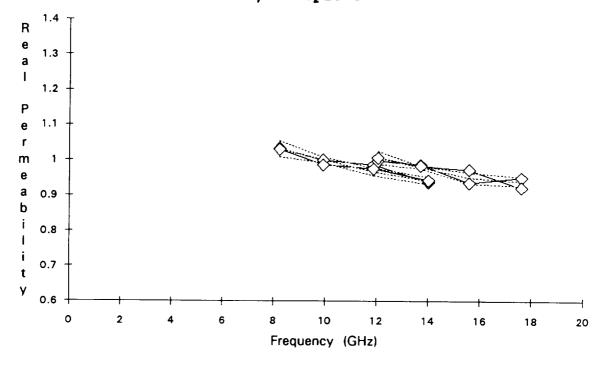


Figure 27. Data from two measurements at each of three frequency bands is plotted vs frequency (solid lines). The average value over all five samples and two measurements at each frequency point is superimposed along with one standard deviation above and below the average (dotted lines).



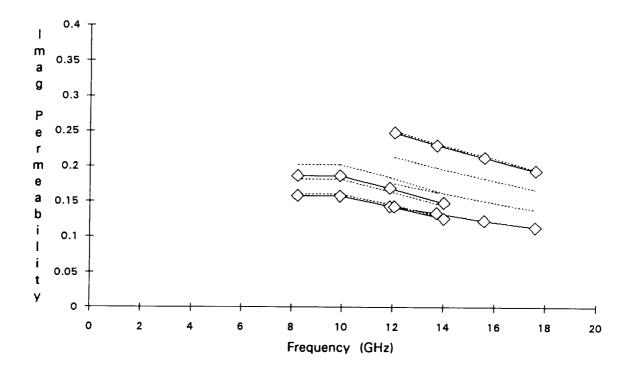


Figure 28. Data from two measurements at each of two frequency bands is plotted vs frequency (solid lines). The average value over all five samples and two measurements at each frequency point is superimposed along with one standard deviation above and below the average (dotted lines).

Batch 2 / Sample 5 1.4 R 1.3 а 1.2 Ρ 1.1 е r m 1 а 0.9 b 0.8 0.7 0.6 0 2 6 10 12 14 16 18 20 Frequency (GHz)

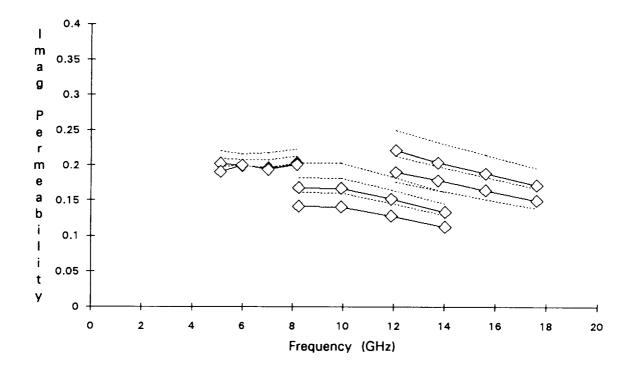


Figure 29. Data from two measurements at each of three frequency bands is plotted vs frequency (solid lines). The average value over all five samples and two measurements at each frequency point is superimposed along with one standard deviation above and below the average (dotted lines).

Batch 3 / Sample 1 1.4 R е 1.3 а 1 1.2 1.1 е r m 1 е а 0.9 b 0.8 0.7 У

10

Frequency (GHz)

12

14

16

18

20

0.6

0

2

6

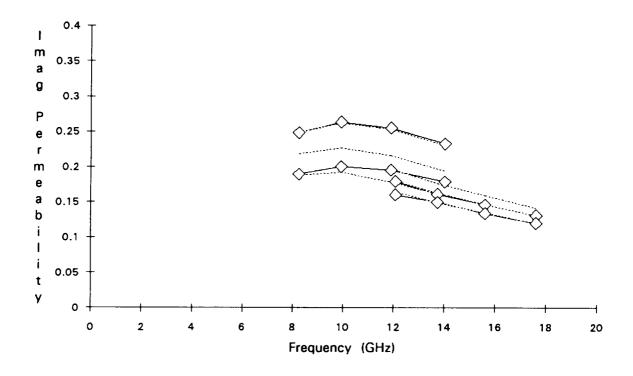
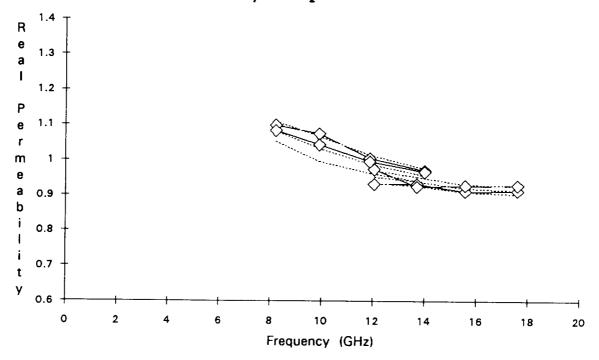


Figure 30. Data from two measurements at each of two frequency bands is plotted vs frequency (solid lines). The average value over all eight samples and two measurements at each frequency point is superimposed along with one standard deviation above and below the average (dotted lines).



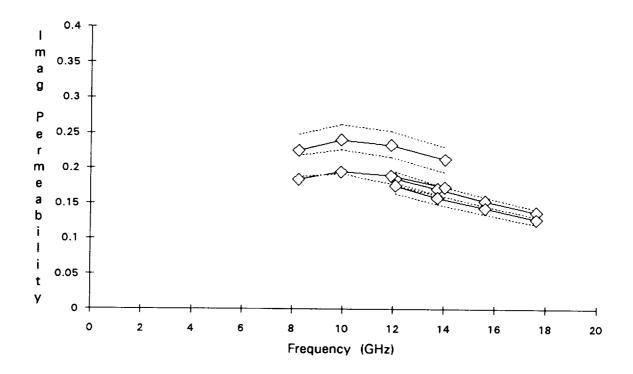
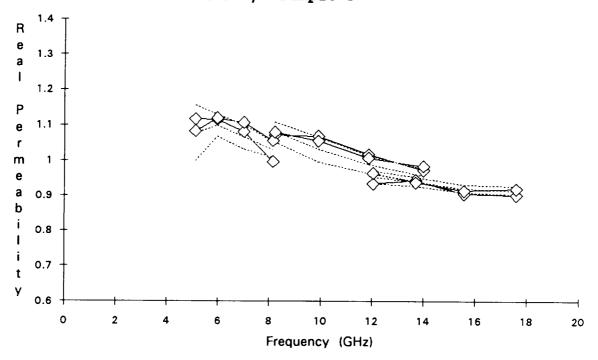


Figure 31. Data from two measurements at each of two frequency bands is plotted vs frequency (solid lines). The average value over all eight samples and two measurements at each frequency point is superimposed along with one standard deviation above and below the average (dotted lines).



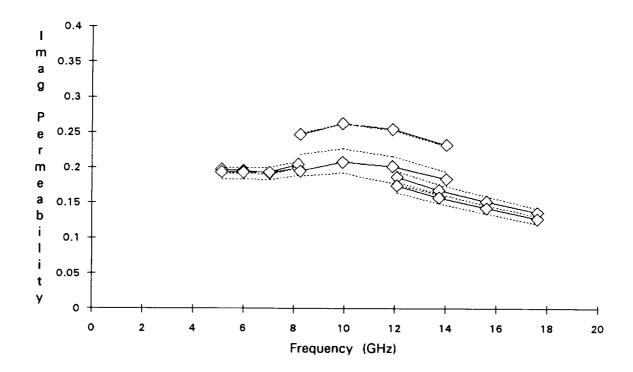
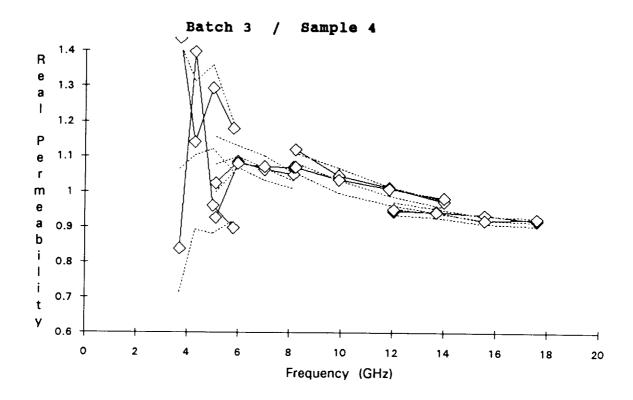


Figure 32. Data from two measurements at each of three frequency bands is plotted vs frequency (solid lines). The average value over all eight samples and two measurements at each frequency point is superimposed along with one standard deviation above and below the average (dotted lines).



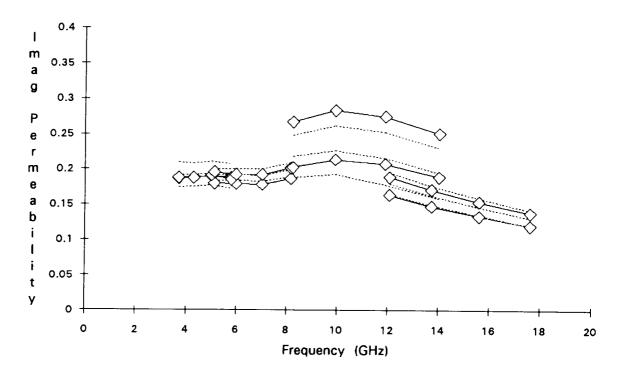
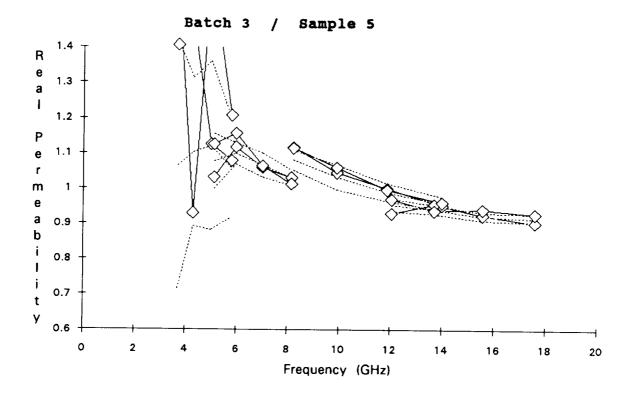


Figure 33. Data from two measurements at each of four frequency bands is plotted vs frequency (solid lines). The average value over all eight samples and two measurements at each frequency point is superimposed along with one standard deviation above and below the average (dotted lines).



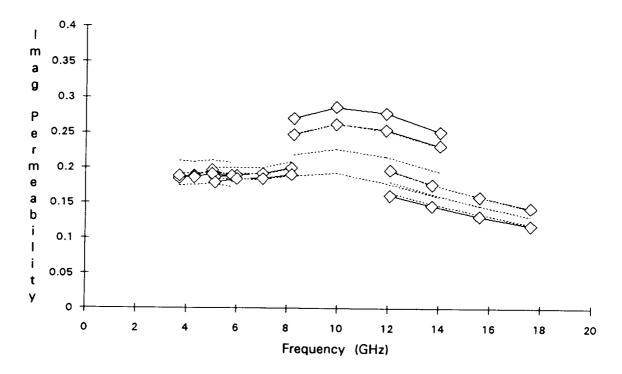
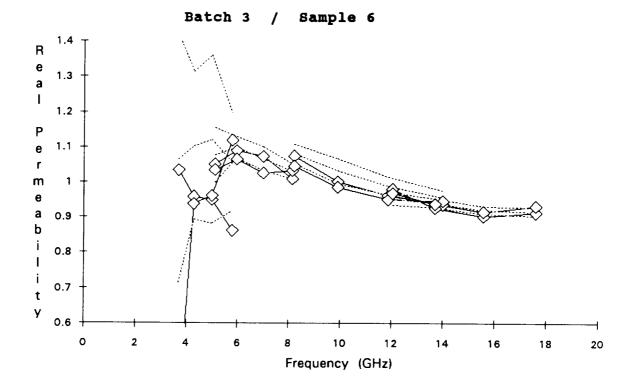


Figure 34 Data from two measurements at each of four frequency bands is plotted vs frequency (solid lines). The average value over all eight samples and two measurements at each frequency point is superimposed along with one standard deviation above and below the average (dotted lines).



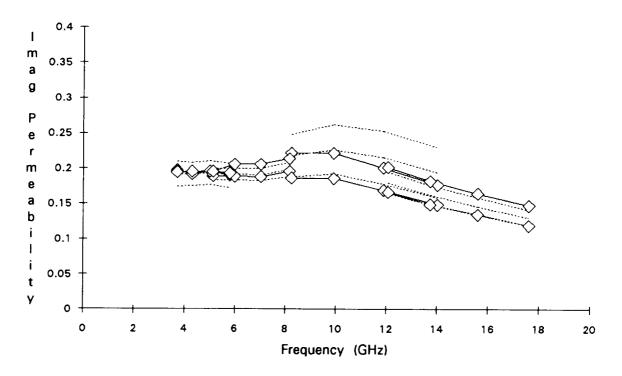
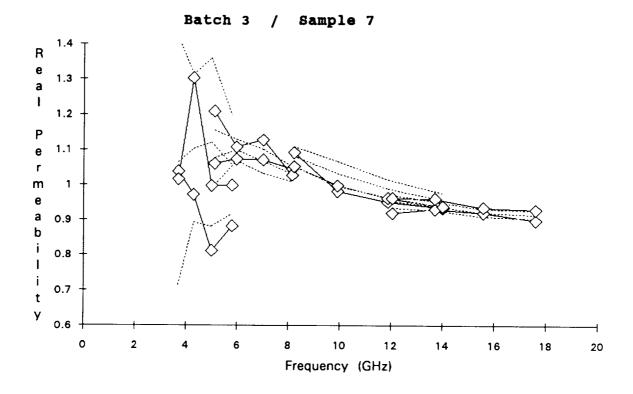


Figure 35 Data from two measurements at each of four frequency bands is plotted vs frequency (solid lines). The average value over all eight samples and two measurements at each frequency point is superimposed along with one standard deviation above and below the average (dotted lines).



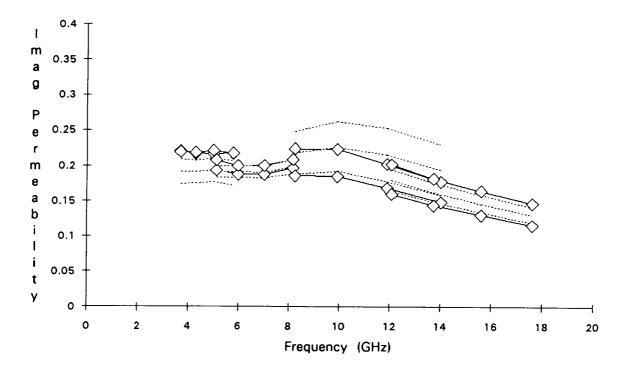
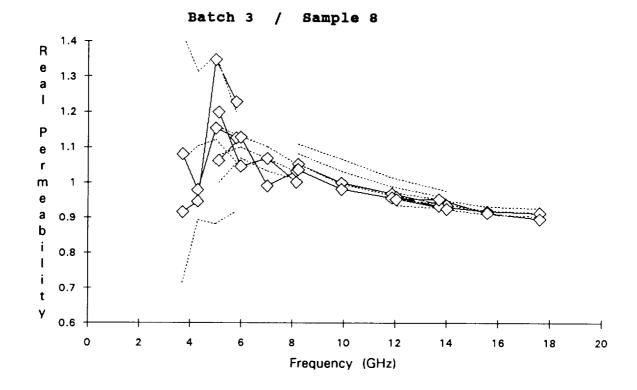


Figure 36 Data from two measurements at each of four frequency bands is plotted vs frequency (solid lines). The average value over all eight samples and two measurements at each frequency point is superimposed along with one standard deviation above and below the average (dotted lines).



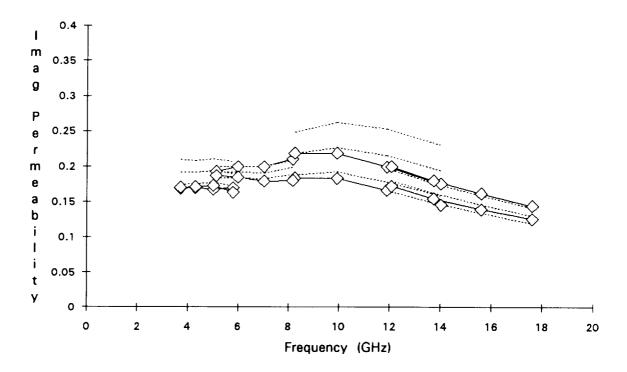


Figure 37 Data from two measurements at each of four frequency bands is plotted vs frequency (solid lines). The average value over all eight samples and two measurements at each frequency point is superimposed along with one standard deviation above and below the average (dotted lines).

D. Error Analysis

The same data points in Figures 2-37 are plotted again in Figures 38-73 with error bars (and without the average and standard deviation lines). This section will present the analysis by which the error bars were obtained. The equations by which the real and imaginary parts of the permittivity are calculated simplify essentially to the following:

$$\varepsilon_r - 1 = \frac{f_0 - f_s}{f_0} \cdot \frac{V_c}{2V_s} = \frac{F_r \cdot V_r}{2}$$

$$\varepsilon_i = \left(\frac{1}{Q_s - Q_0}\right) \cdot \frac{V_c}{4V_s} = \Delta\left(\frac{1}{Q}\right) \cdot \frac{V_r}{4}$$

Where

$$F_r = \frac{f_0 - f_s}{f_0}$$

$$v_r = \frac{v_c}{v_s}$$

$$\Delta\left(\frac{1}{Q}\right) = \left(\frac{1}{Q_s - Q_0}\right)$$

The real and imaginary parts of the permittivity are, of course, represented by ϵ_r and ϵ_i , respectively. The other variables are:

V_C Volume of the cavity

 $V_{\rm S}$ Volume of sample inside the cavity

f₀ Resonant frequency of the empty cavity

 $f_{\rm S}$ Resonant frequency with the sample present

Q₀ Quality factor of the empty cavity

Qs Quality factor with the sample present

The uncertainties in these quantities are related by

$$2 \sigma^{2}(\varepsilon_{r}) = \sigma^{2}(F_{r}) \cdot \left(\frac{\partial \varepsilon_{r}}{\partial F_{r}}\right)^{2} + \sigma^{2}(V_{r}) \cdot \left(\frac{\partial \varepsilon_{r}}{\partial V_{r}}\right)^{2}$$

$$\sigma^{2}(\varepsilon_{r}) = \frac{1}{2}(\sigma^{2}(F_{r}) \cdot V_{r}^{2} + \sigma^{2}(V_{r}) \cdot F_{r}^{2})$$

and

$$4 \sigma^{2}(\varepsilon_{i}) = \sigma^{2}\left(\Delta\left(\frac{1}{Q}\right)\right) \cdot \left(\frac{\partial \varepsilon_{i}}{\partial \Delta\left(\frac{1}{Q}\right)}\right)^{2} + \sigma^{2}(v_{r}) \cdot \left(\frac{\partial \varepsilon_{i}}{\partial v_{r}}\right)^{2}$$

$$\sigma^{2}(\varepsilon_{i}) = \frac{1}{4} \left(\sigma^{2} \left(\Delta \left(\frac{1}{Q} \right) \right) \cdot v_{r}^{2} + \sigma^{2}(v_{r}) \cdot \Delta \left(\frac{1}{Q} \right)^{2} \right)$$

where the standard deviation in a sample population is taken as the uncertainty. It is also necessary that the measurement uncertainty in the volume term be independent of the measurement uncertainty in the frequency term or quality factor term. Then the uncertainty in these terms can be determined from physical measurement considerations.

For the volume ratio term, the uncertainty is derived from

$$\sigma^{2}(v_{r}) = \sigma^{2}(v_{c}) \cdot \left(\frac{\partial v_{r}}{\partial v_{c}}\right)^{2} + \sigma^{2}(v_{s}) \cdot \left(\frac{\partial v_{r}}{\partial v_{s}}\right)^{2}$$

$$= \left(\sigma^{2}(v_{s}) \cdot \left(\frac{v_{c}}{v_{s}}\right)^{2} + \sigma^{2}(v_{c})\right) / v_{s}^{2}$$

The cavity volume contributes

$$\sigma^{2}(V_{c}) = \sigma^{2}(a)(b\ell)^{2} + \sigma^{2}(b)(a\ell)^{2} + \sigma^{2}(\ell)(ab)^{2}$$

where the uncertainty in the transverse dimensions, a and b, are the mechanical tolerances of the waveguide, and the measurement uncertainty of 0.08 cm (0.03 in.) in the length is used for $\sigma(\ell)$

The volume of sample in the cavity is calculated for each measurement using the appropriate transverse waveguide dimension (broad wall for permeability measurements and narrow wall for permittivity measurements) and the cross sectional area of the individual sample. The sample cross section is assumed to be uniform and is calculated using the density values supplied with the samples after weighing each sample and measuring its length. Then the sample volume uncertainty is

$$\sigma^{2}(v_{s}) = \sigma^{2}(x)(\ell_{s})^{2} + \sigma^{2}(\ell_{s})(x)^{2}$$

where ℓ_s is the length of sample in the cavity and x is the cross section area. Of particular concern is the effect of the value used for the density of a sample material. The precision of the density value supplied with the samples was taken to be $\pm .01$ g/cc. With this precision for the density, the uncertainty in the sample volume is dominated by the uncertainty in the measured length of the sample, which was determined to be .08 cm (.03 in).

The frequency ratio term $F_{\mathbf{r}}$ contributes to the measurement uncertainty as follows:

$$\sigma^{2}(F_{r}) = \sigma^{2}(f_{0}) \cdot \left(\frac{\partial F_{r}}{\partial f_{0}}\right)^{2} + \sigma^{2}(f_{s}) \cdot \left(\frac{\partial F_{r}}{\partial f_{s}}\right)^{2}$$

$$= \sigma^{2}(f_{0}) \cdot \left(\frac{f_{s}}{f_{0}^{2}}\right)^{2} + \sigma^{2}(f_{s}) \cdot \left(\frac{1}{f_{0}^{2}}\right)^{2}$$

Here the variance terms are computed from the measurement results. Specifically, the variance term for the empty cavity resonance, f_0 , is calculated as the sample variance for the population of empty cavity resonant frequency values recorded at each mode with each batch of samples. The variance term for the perturbed resonant frequency, $f_{\rm S}$, is likewise calculated from the measurement population at each mode for each batch of samples. Note that this means that sample to sample variation within a batch, as well as inhomogeneity within each sample, will contribute to the measurement uncertainty, and this is accounted for in the plotted error bars. Similarly, uncertainty in the quality factor measurements is taken as the measurement variance at each mode for each batch of samples, and contributes to the

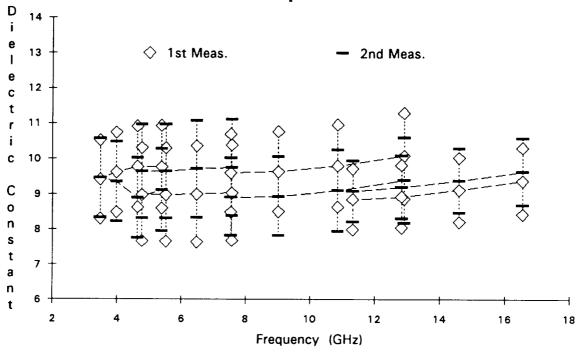
overall measurement uncertainty through the $^{\Delta}\left(\frac{1}{Q}\right)$ term as follows:

$$\sigma^{2}\left(\left(\Delta\left(\frac{1}{Q}\right)\right)\right) = \sigma^{2}\left(\frac{1}{Q_{s}}\right) \cdot \left(\frac{\partial\left(\Delta\left(\frac{1}{Q}\right)\right)}{\partial\left(\frac{1}{Q_{s}}\right)}\right)^{2} + \sigma^{2}\left(\frac{1}{Q_{0}}\right) \cdot \left(\frac{\partial\left(\Delta\left(\frac{1}{Q}\right)\right)}{\partial\left(\frac{1}{Q_{0}}\right)}\right)^{2}$$

$$= \sigma^{2}\left(\frac{1}{Q_{s}}\right) + \sigma^{2}\left(\frac{1}{Q_{0}}\right)$$

Again, the effects of sample to sample variation within a sample batch, and the effects of inhomogeneity within a particular sample, have been accounted for in the error bars for these terms.

Plots of the variations in the measured quantities at each resonant mode have been included in the appendix as a quick visual indication of the magnitude of sample-to-sample variations. For these plots, the empty cavity quantities have been plotted as deviations from the average value, where the average is computed across all empty cavity measurements at a given frequency mode. For the top plot on each page the plotted quantity is a frequency deviation, while the bottom plot shows variations in the quantity 1/Q. The dashed lines in each plot represent the associated with the introduction of a sample into the Thus the solid lines indicate the magnitude of the standard deviations used for f_0 and $1/Q_0$ in the error bar calculations, while the dashed lines show the relative magnitude of the standard deviations of the quantities f_s and $1/Q_{S}$.



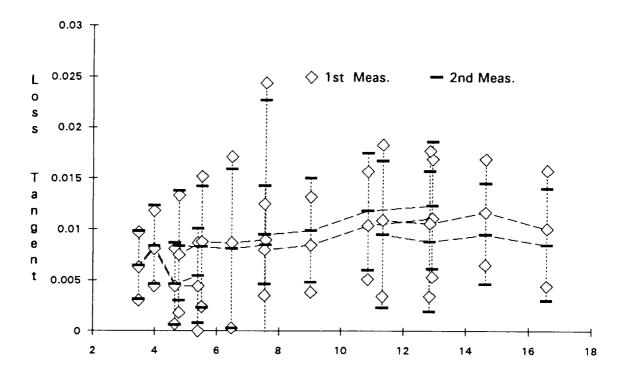
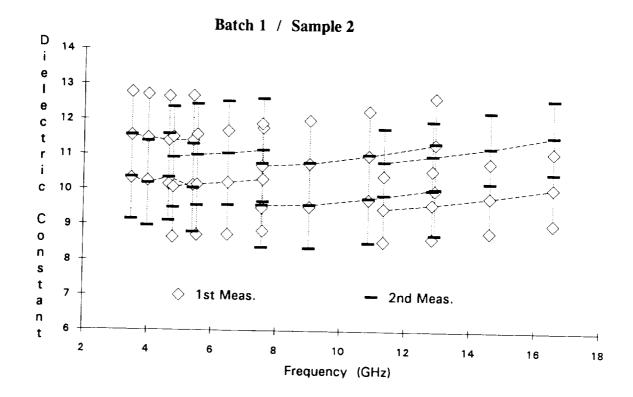


Figure 38. The data from Figure 2 is plotted here with error bars around each point. The error bars represent a calculated uncertainty level of one standard deviation as explained in the text. They include the effect of variations from sample to sample within the same batch.



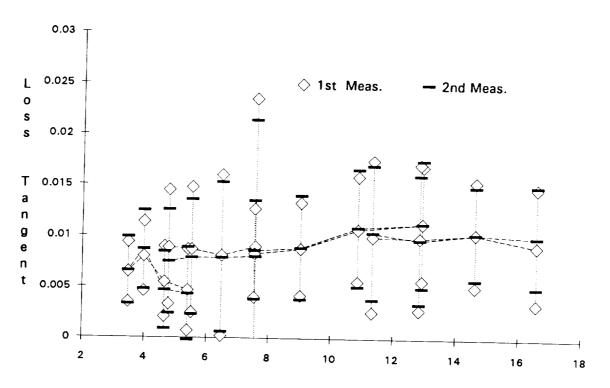
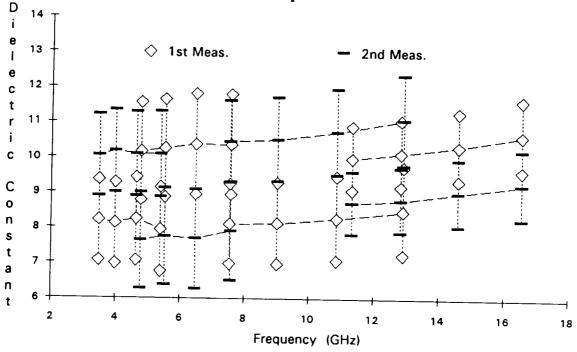


Figure 39. The data from Figure 3 is plotted here with error bars around each point. The error bars represent a calculated uncertainty level of one standard deviation as explained in the text. They include the effect of variations from sample to sample within the same batch.



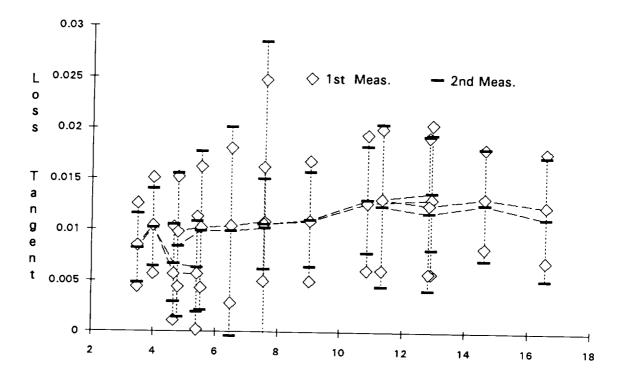
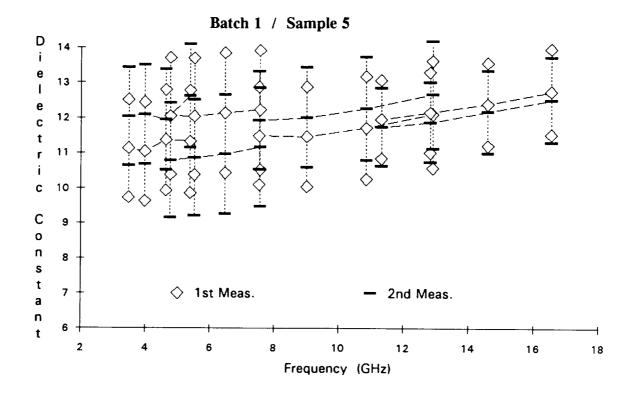


Figure 40. The data from Figure 4 is plotted here with error bars around each point. The error bars represent a calculated uncertainty level of one standard deviation as explained in the text. They include the effect of variations from sample to sample within the same batch.



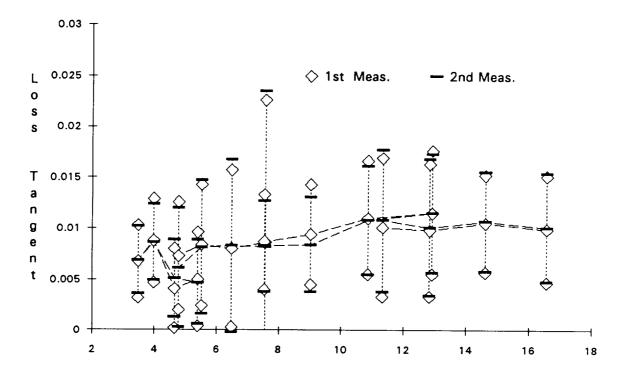


Figure 41. The data from Figure 5 is plotted here with error bars around each point. The error bars represent a calculated uncertainty level of one standard deviation as explained in the text. They include the effect of variations from sample to sample within the same batch.

Batch 1 / Sample 8 14 i 13 е 12 С t 11 ſ i C 10 С 9 0 n 8 t 7 1st Meas. 2nd Meas. а 2 10 12 14 16 18 Frequency (GHz)

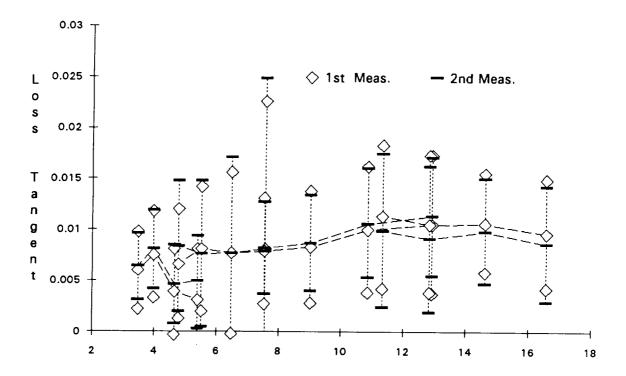
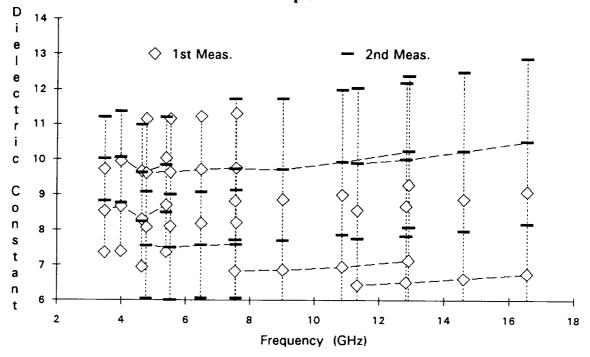


Figure 42. The data from Figure 6 is plotted here with error bars around each point. The error bars represent a calculated uncertainty level of one standard deviation as explained in the text. They include the effect of variations from sample to sample within the same batch.



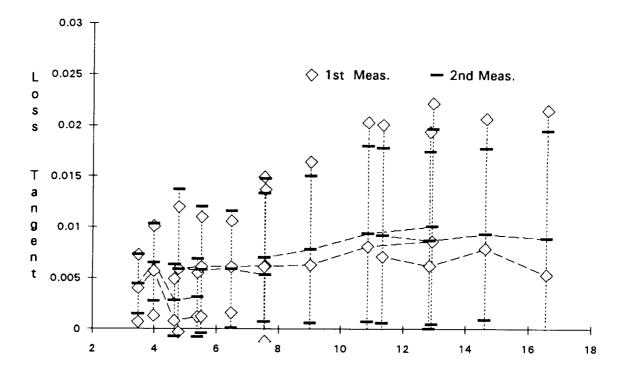
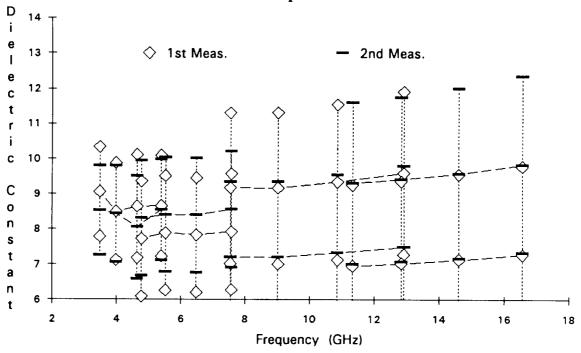


Figure 43. The data from Figure 7 is plotted here with error bars around each point. The error bars represent a calculated uncertainty level of one standard deviation as explained in the text. They include the effect of variations from sample to sample within the same batch.



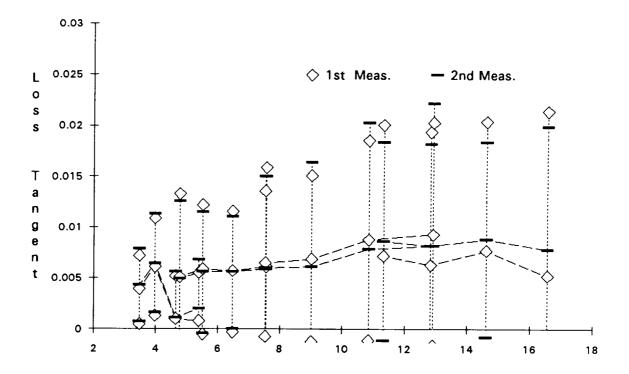
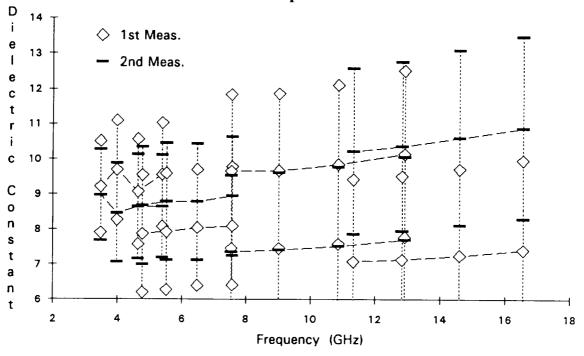


Figure 44. The data from Figure 8 is plotted here with error bars around each point. The error bars represent a calculated uncertainty level of one standard deviation as explained in the text. They include the effect of variations from sample to sample within the same batch.



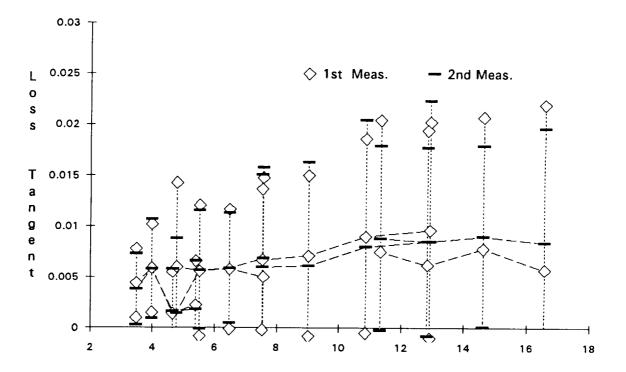
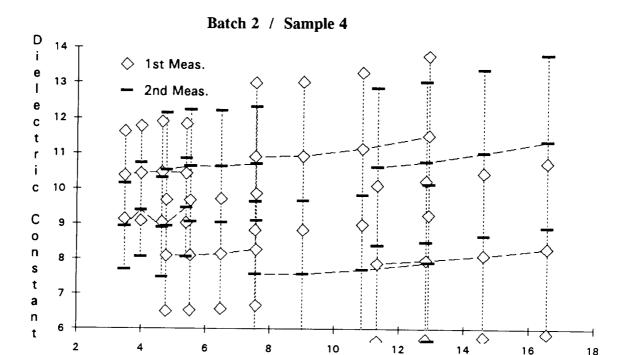


Figure 45. The data from Figure 9 is plotted here with error bars around each point. The error bars represent a calculated uncertainty level of one standard deviation as explained in the text. They include the effect of variations from sample to sample within the same batch.



Frequency (GHz)

16

18

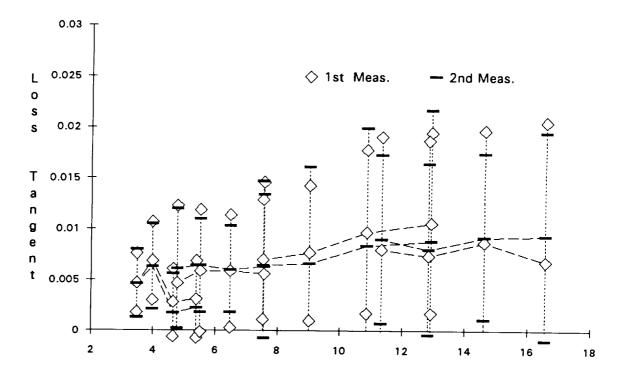
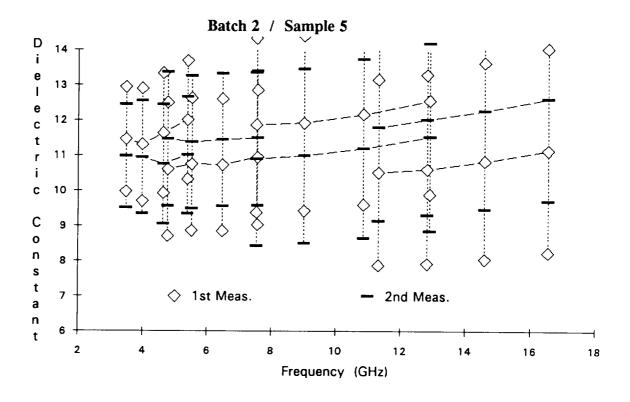


Figure 46. The data from Figure 10 is plotted here with error bars around each point. The error bars represent a calculated uncertainty level of one standard deviation as explained in the text. They include the effect of variations from sample to sample within the same batch.



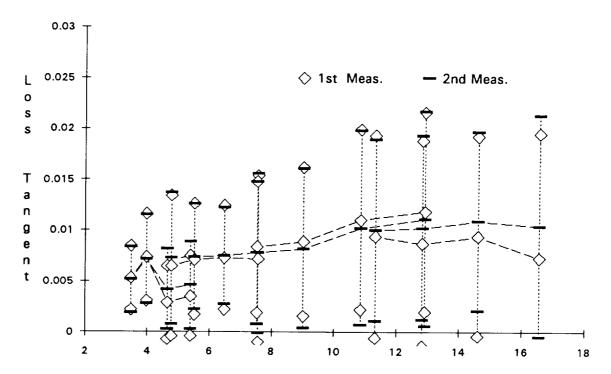
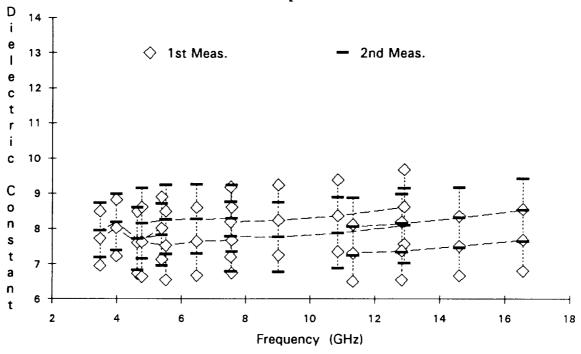


Figure 47. The data from Figure 11 is plotted here with error bars around each point. The error bars represent a calculated uncertainty level of one standard deviation as explained in the text. They include the effect of variations from sample to sample within the same batch.



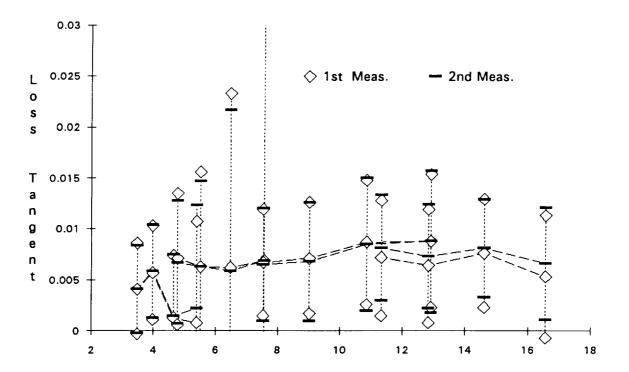
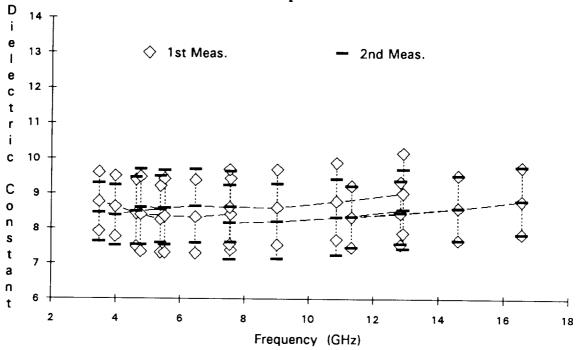


Figure 48. The data from Figure 12 is plotted here with error bars around each point. The error bars represent a calculated uncertainty level of one standard deviation as explained in the text. They include the effect of variations from sample to sample within the same batch.



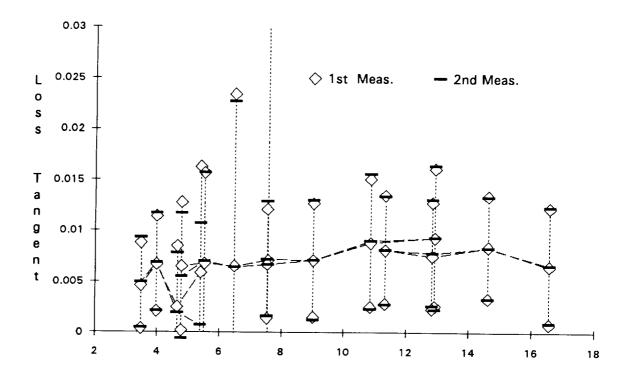
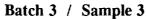
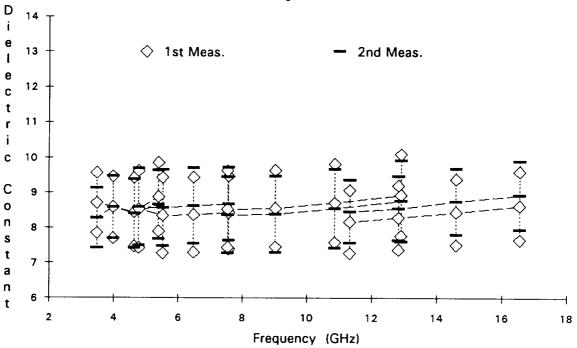


Figure 49. The data from Figure 13 is plotted here with error bars around each point. The error bars represent a calculated uncertainty level of one standard deviation as explained in the text. They include the effect of variations from sample to sample within the same batch.





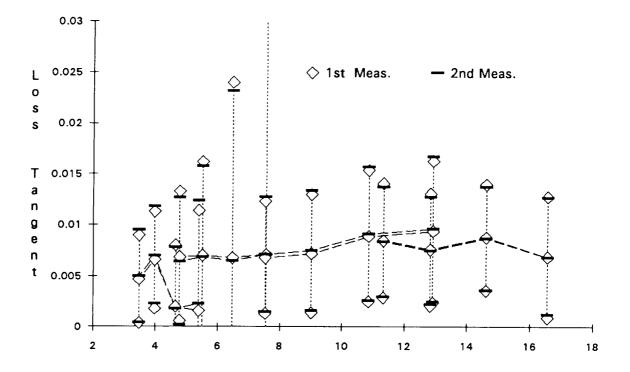
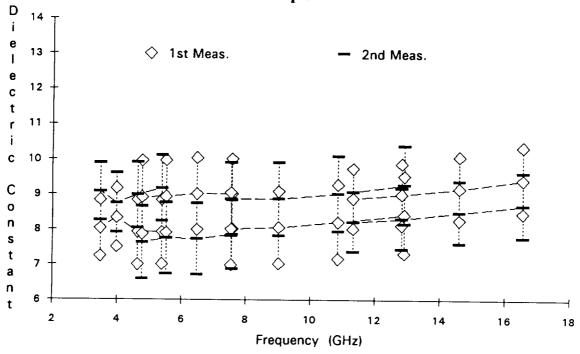


Figure 50. The data from Figure 14 is plotted here with error bars around each point. The error bars represent a calculated uncertainty level of one standard deviation as explained in the text. They include the effect of variations from sample to sample within the same batch.



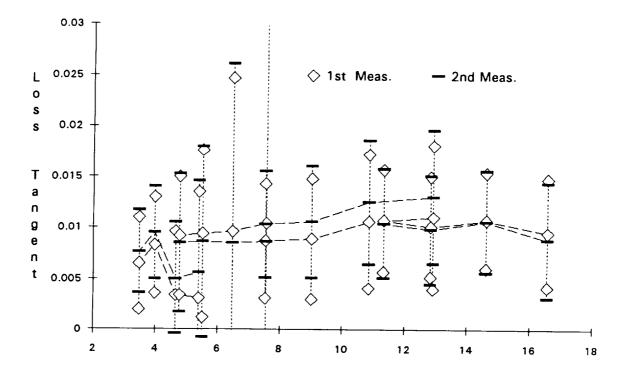
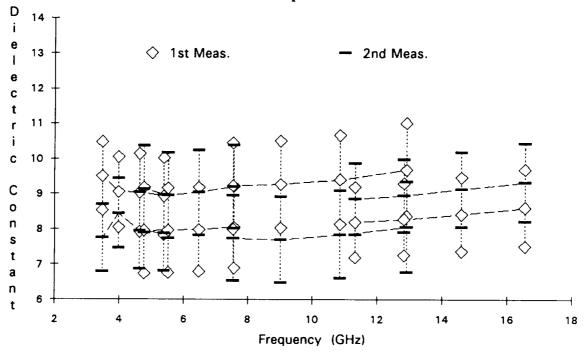


Figure 51. The data from Figure 15 is plotted here with error bars around each point. The error bars represent a calculated uncertainty level of one standard deviation as explained in the text. They include the effect of variations from sample to sample within the same batch.



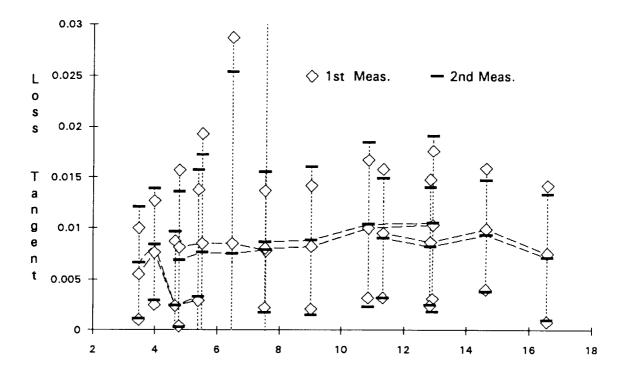
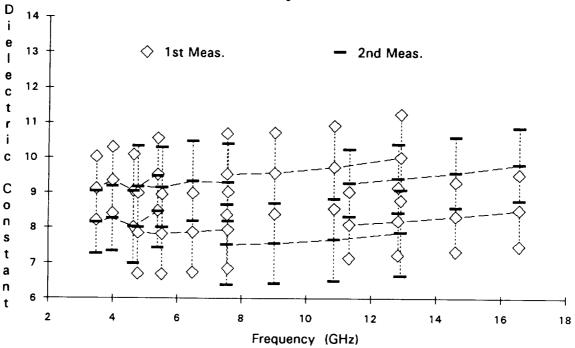


Figure 52. The data from Figure 16 is plotted here with error bars around each point. The error bars represent a calculated uncertainty level of one standard deviation as explained in the text. They include the effect of variations from sample to sample within the same batch.



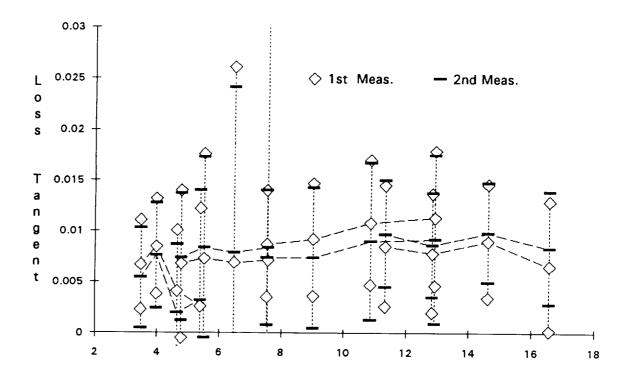
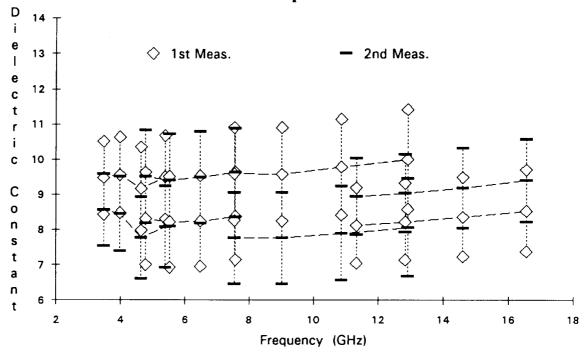


Figure 53. The data from Figure 17 is plotted here with error bars around each point. The error bars represent a calculated uncertainty level of one standard deviation as explained in the text. They include the effect of variations from sample to sample within the same batch.



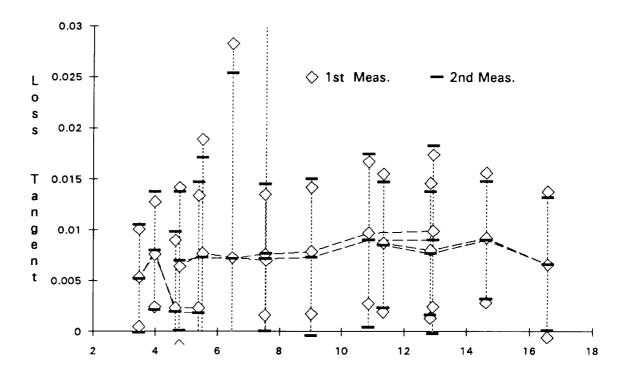
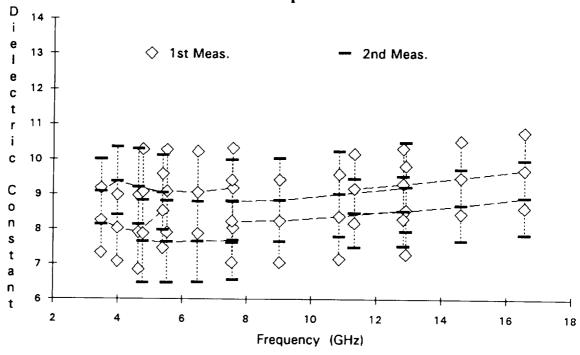


Figure 54. The data from Figure 18 is plotted here with error bars around each point. The error bars represent a calculated uncertainty level of one standard deviation as explained in the text. They include the effect of variations from sample to sample within the same batch.



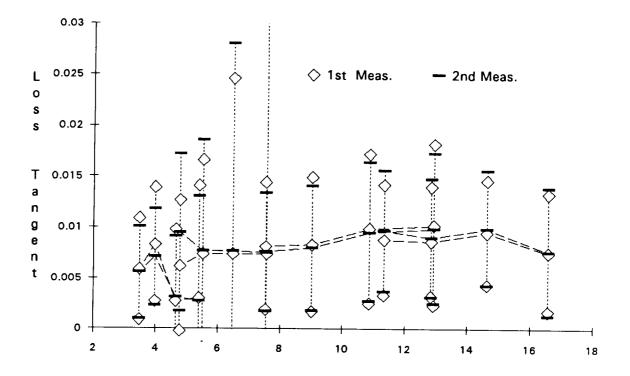
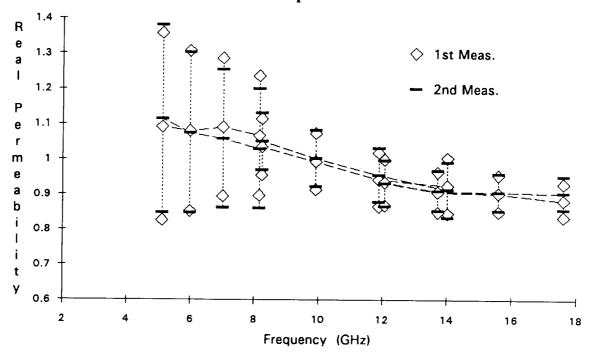


Figure 55. The data from Figure 19 is plotted here with error bars around each point. The error bars represent a calculated uncertainty level of one standard deviation as explained in the text. They include the effect of variations from sample to sample within the same batch.



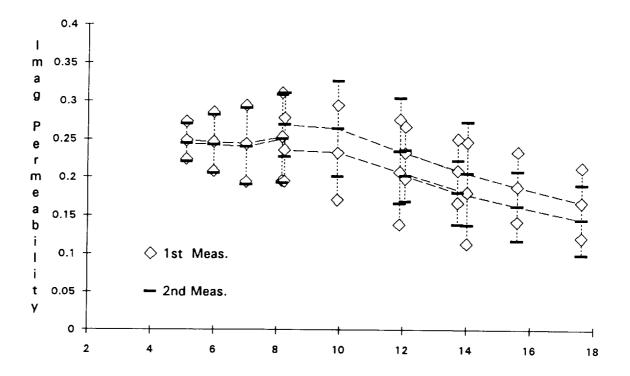
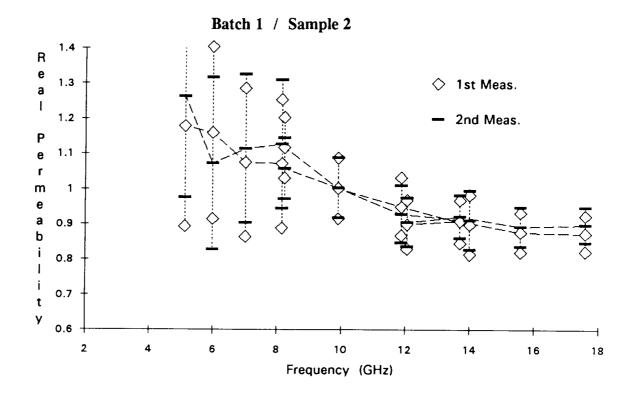


Figure 56. The data from Figure 20 is plotted here with error bars around each point. The error bars represent a calculated uncertainty level of one standard deviation as explained in the text. They include the effect of variations from sample to sample within the same batch.



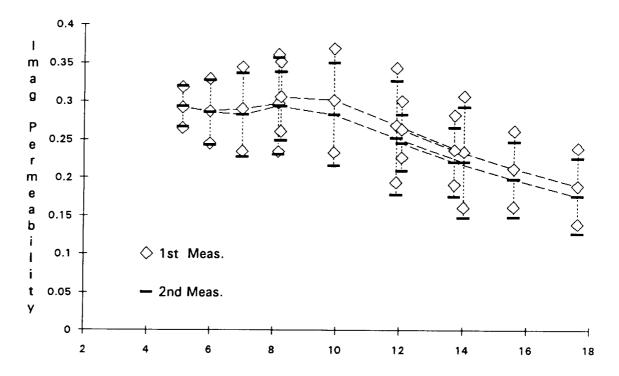
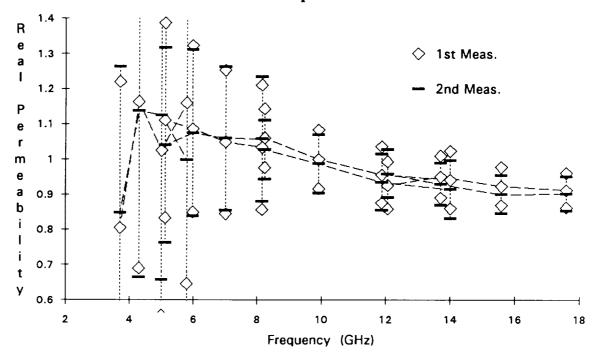


Figure 57. The data from Figure 21 is plotted here with error bars around each point. The error bars represent a calculated uncertainty level of one standard deviation as explained in the text. They include the effect of variations from sample to sample within the same batch.

Batch 1 / Sample 4



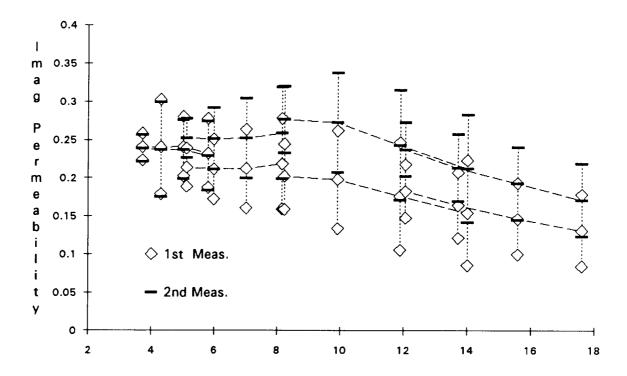
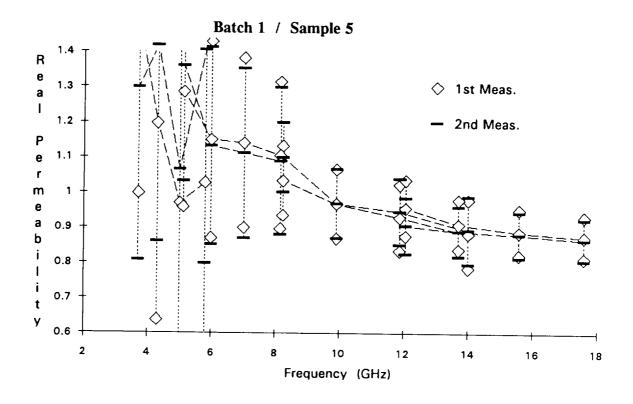


Figure 58. The data from Figure 22 is plotted here with error bars around each point. The error bars represent a calculated uncertainty level of one standard deviation as explained in the text. They include the effect of variations from sample to sample within the same batch.



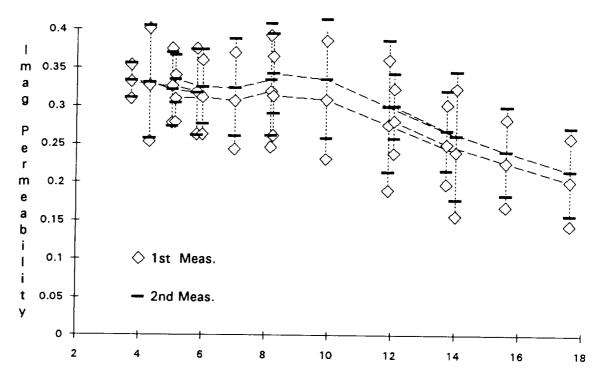
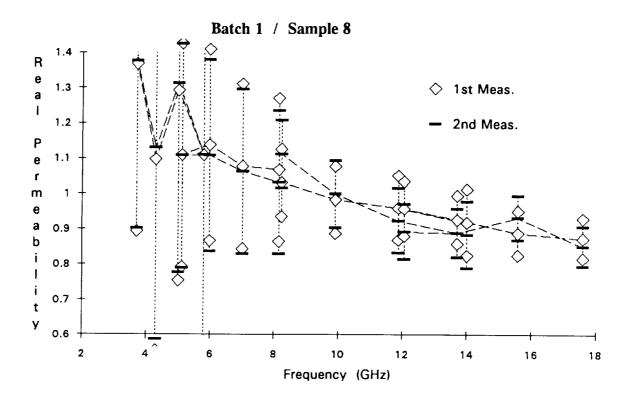


Figure 59. The data from Figure 23 is plotted here with error bars around each point. The error bars represent a calculated uncertainty level of one standard deviation as explained in the text. They include the effect of variations from sample to sample within the same batch.



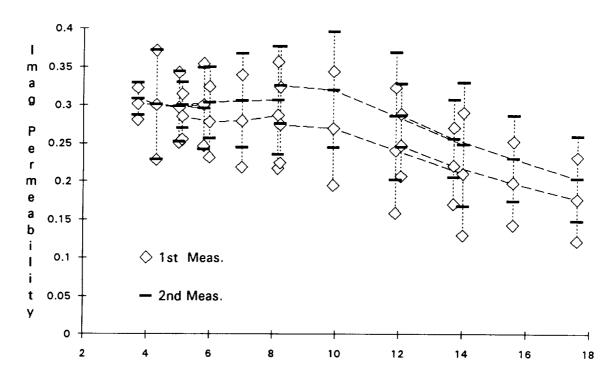
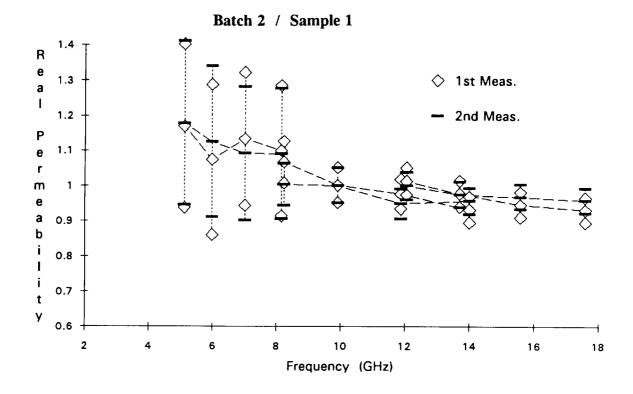


Figure 60. The data from Figure 24 is plotted here with error bars around each point. The error bars represent a calculated uncertainty level of one standard deviation as explained in the text. They include the effect of variations from sample to sample within the same batch.



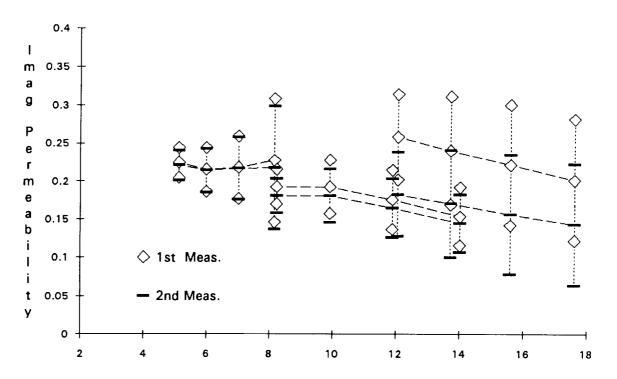
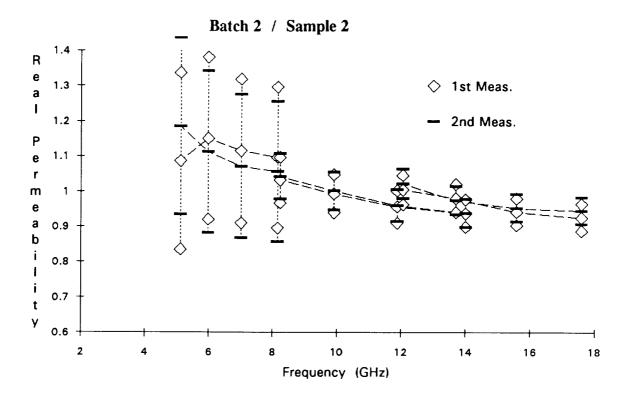


Figure 61. The data from Figure 25 is plotted here with error bars around each point. The error bars represent a calculated uncertainty level of one standard deviation as explained in the text. They include the effect of variations from sample to sample within the same batch.



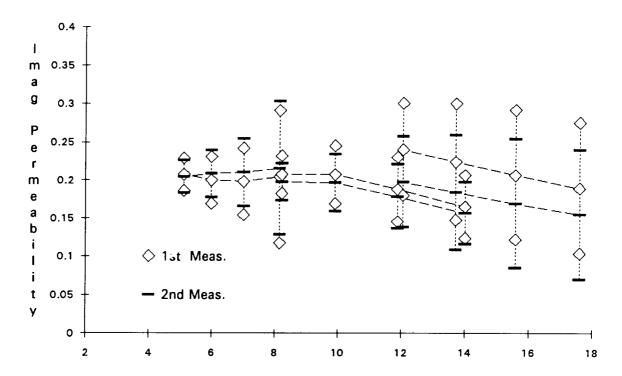
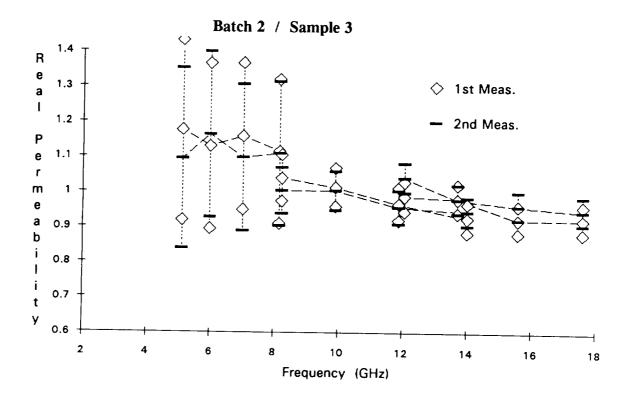


Figure 62. The data from Figure 26 is plotted here with error bars around each point. The error bars represent a calculated uncertainty level of one standard deviation as explained in the text. They include the effect of variations from sample to sample within the same batch.



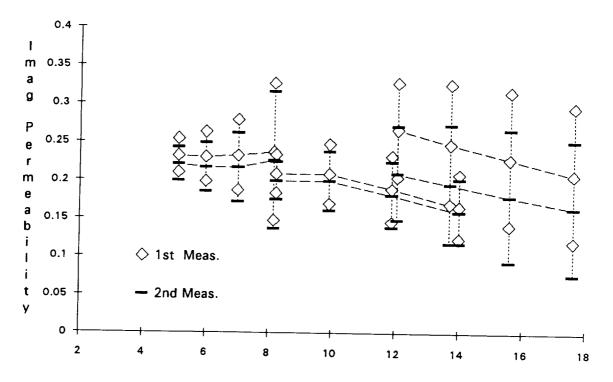
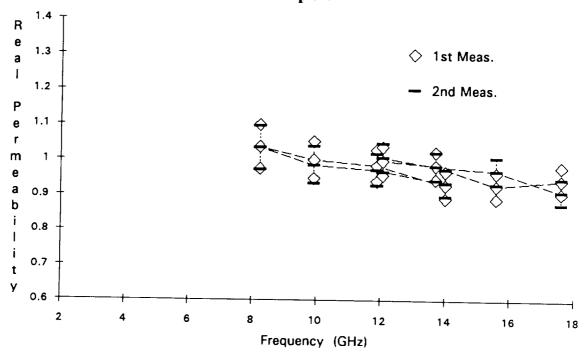


Figure 63. The data from Figure 27 is plotted here with error bars around each point. The error bars represent a calculated uncertainty level of one standard deviation as explained in the text. They include the effect of variations from sample to sample within the same batch.

Batch 2 / Sample 4



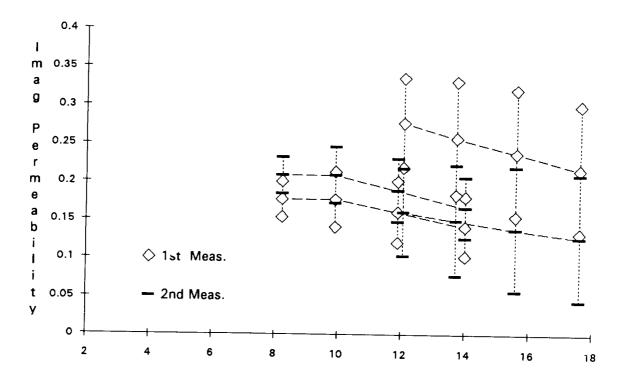
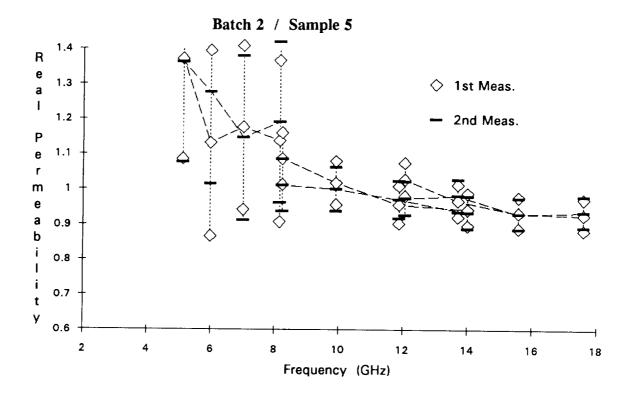


Figure 64. The data from Figure 28 is plotted here with error bars around each point. The error bars represent a calculated uncertainty level of one standard deviation as explained in the text. They include the effect of variations from sample to sample within the same batch.



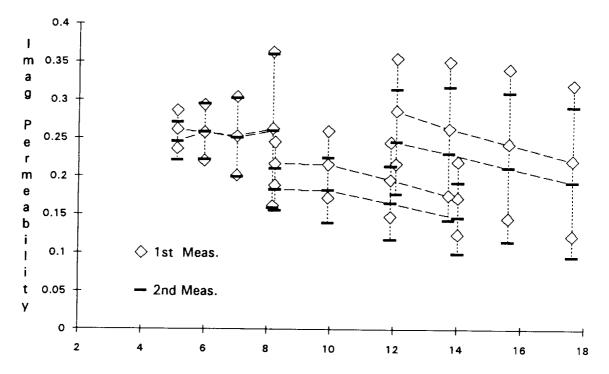
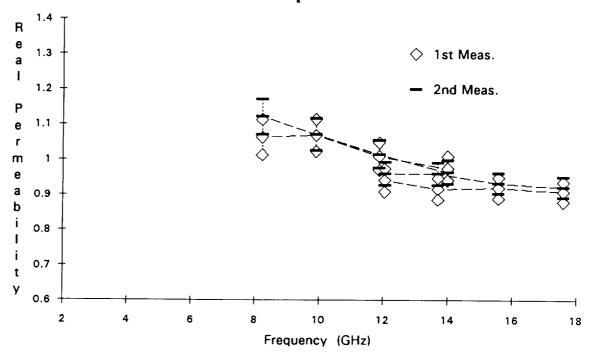


Figure 65. The data from Figure 29 is plotted here with error bars around each point. The error bars represent a calculated uncertainty level of one standard deviation as explained in the text. They include the effect of variations from sample to sample within the same batch.

Batch 3 / Sample 1



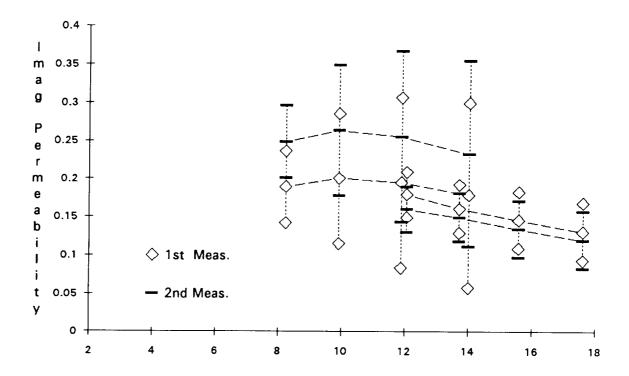
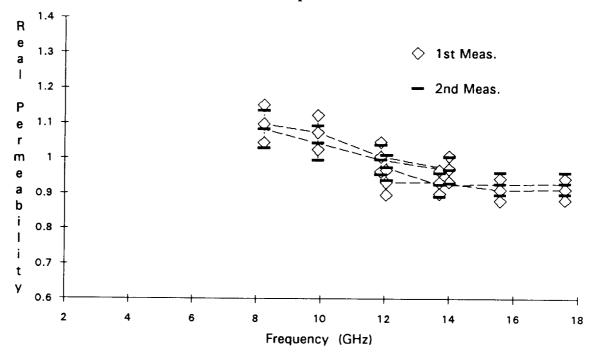


Figure 66. The data from Figure 30 is plotted here with error bars around each point. The error bars represent a calculated uncertainty level of one standard deviation as explained in the text. They include the effect of variations from sample to sample within the same batch.

Batch 3 / Sample 2



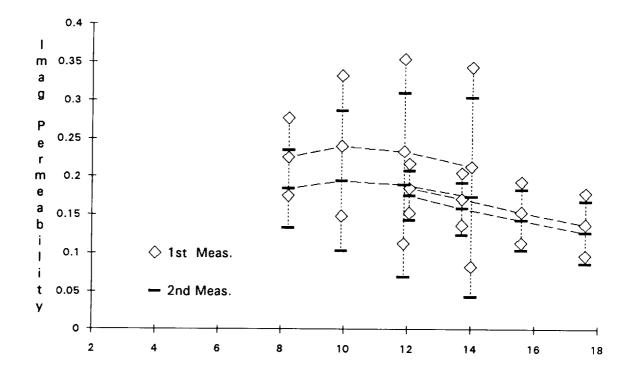
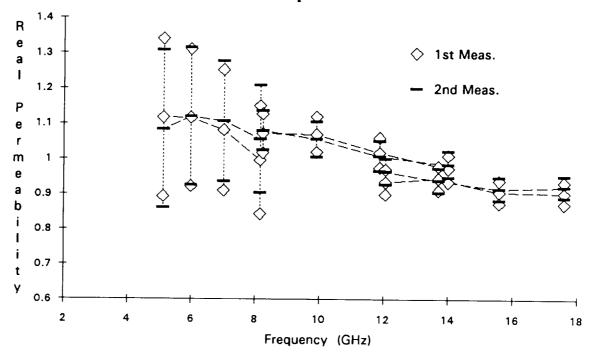


Figure 67. The data from Figure 31 is plotted here with error bars around each point. The error bars represent a calculated uncertainty level of one standard deviation as explained in the text. They include the effect of variations from sample to sample within the same batch.

Batch 3 / Sample 3



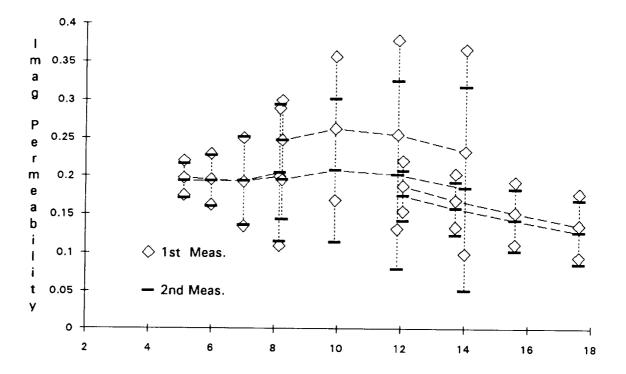
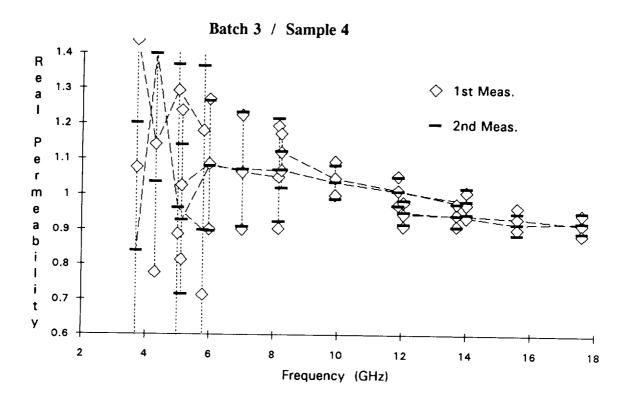


Figure 68. The data from Figure 32 is plotted here with error bars around each point. The error bars represent a calculated uncertainty level of one standard deviation as explained in the text. They include the effect of variations from sample to sample within the same batch.



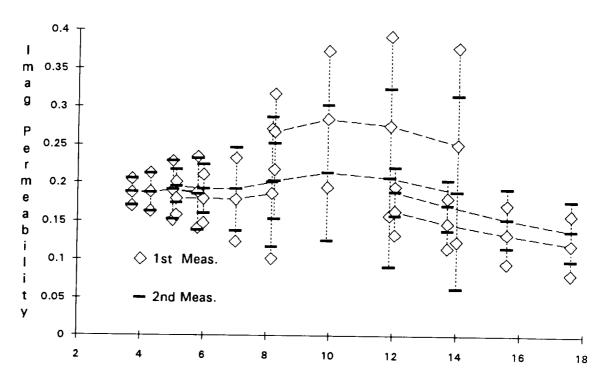
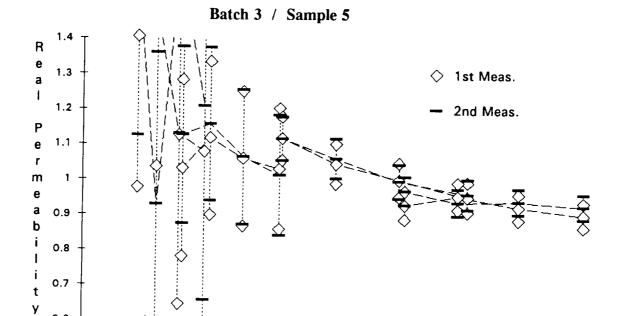


Figure 69. The data from Figure 33 is plotted here with error bars around each point. The error bars represent a calculated uncertainty level of one standard deviation as explained in the text. They include the effect of variations from sample to sample within the same batch.



Frequency (GHz)

0.6

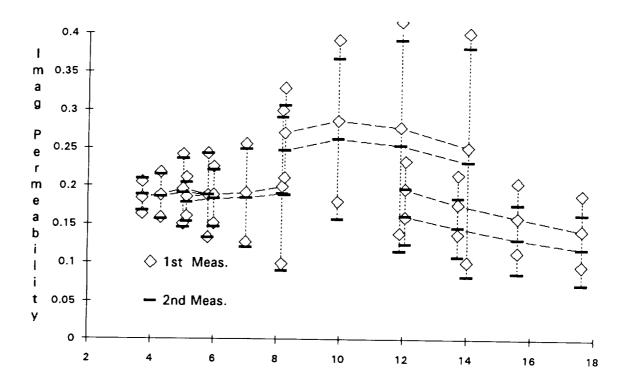
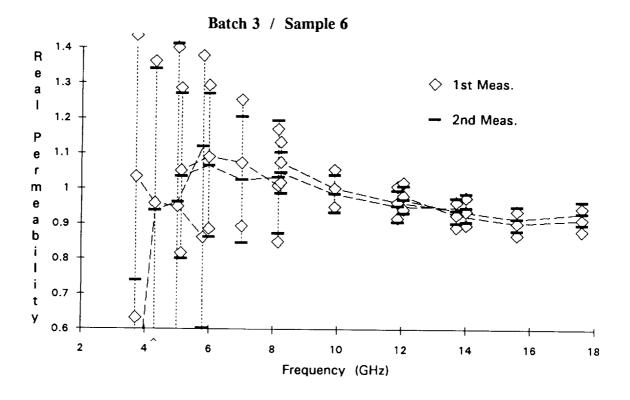


Figure 70. The data from Figure 34 is plotted here with error bars around each point. The error bars represent a calculated uncertainty level of one standard deviation as explained in the text. They include the effect of variations from sample to sample within the same batch.



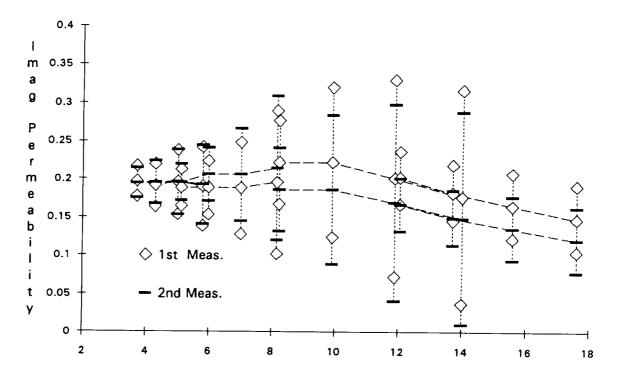
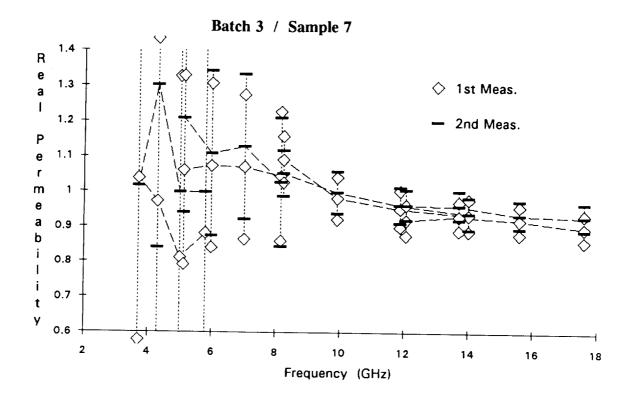


Figure 71. The data from Figure 35 is plotted here with error bars around each point. The error bars represent a calculated uncertainty level of one standard deviation as explained in the text. They include the effect of variations from sample to sample within the same batch.



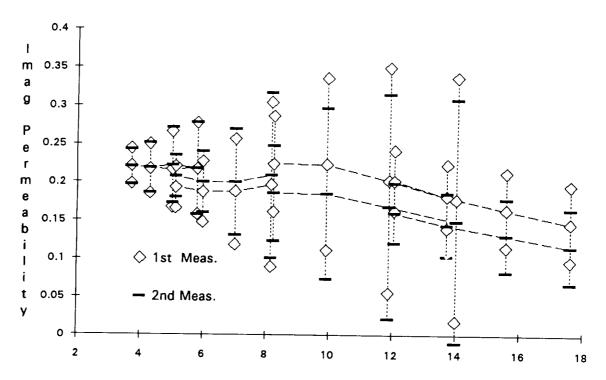
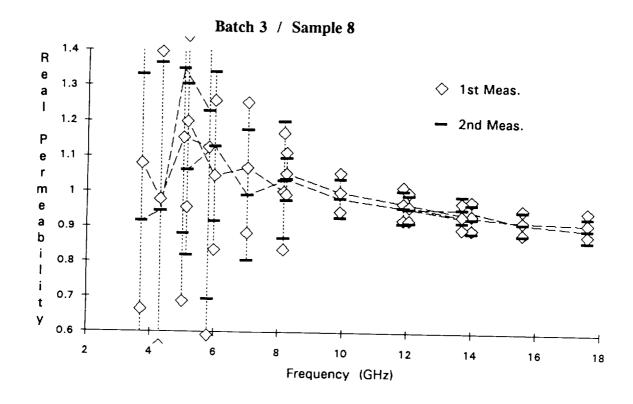


Figure 72. The data from Figure 36 is plotted here with error bars around each point. The error bars represent a calculated uncertainty level of one standard deviation as explained in the text. They include the effect of variations from sample to sample within the same batch.



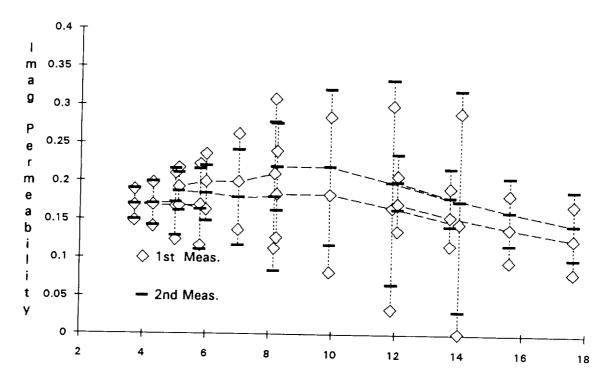


Figure 73. The data from Figure 37 is plotted here with error bars around each point. The error bars represent a calculated uncertainty level of one standard deviation as explained in the text. They include the effect of variations from sample to sample within the same batch.

III. Financial Status

Of the total grant of \$100,030, expenditures through December 31 have totalled \$32,640, or approximately 33%.

IV. References

- American Society for Testing and Materials standard D 2520-86, "Complex Permittivity of Solid Electrical Insulating Materials at Microwave Frequencies and Temperatures to 1650°C."
- American Society for Testing and Materials standard F 131-70, "Complex Dielectric Constant of Nonmetallic Magnetic Materials at Microwave Frequencies."
- Moore, et al., "Second Order Correction in Cavity Constitutive Parameter Measurements with Application to Anisotropic Ferrites," International Symposium on Infrared and MMW Technology, Orlando, Florida, December, 1987.

APPENDIX A

The plots in this appendix give an indication of the repeatability of the measurements. A solid line represents a quantity in the unperturbed cavity for the mode indicated in the plot title. In all of the plots, a solid line represents the deviation from the average value of the empty cavity resonant frequency, f₀, or the empty cavity quality factor, as represented by the quantity $1/Q_0$. smoothness of the solid lines is an indication of the repeatability of empty cavity measurements, and consequently of the calculated quantities. The dashed line represents perturbation caused in a given mode by the presence of a For odd modes, the difference between the solid sample. line and dashed line in the frequency plot would be the quantity used to calculate the dielectric constant. The difference in the quality factor plot would be used to calculate the imaginary part of the permittivity. The loss tangent is just the ratio of imaginary and Similarly, the difference between solid and permittivity. dashed lines in the frequency plot for even modes is used to calculate the real part of the permeability, while the difference in the quality factor plot is used to calculate the imaginary part. The smoother a line is, the less variation one would see in calculated parameters. The three sets of lines in each plot represent results from the three different batches of samples. The abscissa is meant to indicate the chronological order of the measurements rather than any fixed time increment, with one unit representing one measurement.

